

# THE B.D.H. BOOK OF A.R. STANDARDS

# THE RDH BOOK

# THE B.D.H. BOOK OF A.R. STANDARDS (Second Edition)

# CORRIGENDA

Page 1, line 11, for 0.04 read 0.009

Page 9, line 14, for 1 gram dissolved in 50 c.c. of water read 1 gram with 1 gram of sodium hydroxide (NH<sub>3</sub> free) dissolved in 50 c.c. of water

Page 13, line 4, for 0.02 read 0.01

Page 13, line 12, for 5 grams read 10 grams

Page 15, line 2, for 6 grams of citric acid read 7 grams of citric acid

Page 31, after line 5, insert Arsenic (As,O3) . . . 0.0005 per cent,

Page 31, after line 12 insert Arsenic Limit 5 parts per million

Page 69, line 9, for (calculated as H<sub>2</sub>O<sub>2</sub>) read (calculated as ether peroxide)

Page 83, line 25, for 0.00004 read 0.000004

Page 97, line 11, before orange insert yellow or

Page 130, line 35, for K2SO3 read K2CO3

Page 152, lines 14 and 15, to read as follows: add 25 c.c. of water, 3 grams of potassium iodide and 15 c.c. of hydrochloric acid and titrate the liberated iodine immediately

Page 153, line 25, for 157° read 158°

Page 181, line 30, after water insert , cool to  $60^{\circ}$ 

THE BRITISH DRUG HOUSES LTD.

April 1933

# THE B.D.H. BOOK OF A.R. STANDARDS



Second and Revised Edition

THE BRITISH DRUG HOUSES LTD. LONDON N.1

# PREFATORY NOTE TO SECOND EDITION

Six years have elapsed since the issue of the first edition of *The B.D.H. Book of A.R. Standards*. During this period important advances in analytical practice have been made, and new and delicate tests have been devised. Many of the B.D.H. specifications have been made more stringent, others have been more accurately defined, and in the new and revised edition of *The B.D.H. Book of A.R. Standards*, now being issued, it has been felt desirable to include an even wider range of chemicals to which the term "A.R." can be applied.

The second edition of *The B.D.H. Book of A.R. Standards* contains revised monographs for the 158 chemicals contained in the first edition, and also new monographs for 50 other substances. In addition, for the convenience of users, the limiting values of the various tests have been stated at the head of each monograph in the form of a table of maximum permissible limits of impurities.

Suggestions from users of this book for the further improvement of the text will be greatly appreciated.

THE BRITISH DRUG HOUSES LTD.

July, 1932.

# PREFATORY NOTE TO FIRST EDITION

IN 1914, when the supply of German analytical reagents was cut off by the war, the Institute of Chemistry and the Society of Public Analysts stepped into the breach and performed a highly useful public service. A Joint Committee was formed from the Councils of these two bodies, which Committee drew up and issued a list of specifications of purity for 88 analytical reagents. These specifications of purity were indicated by the letters "A.R.", which have since become well known to British chemists.

The British Drug Houses Ltd. undertook the manufacture of analytical reagents at this time, opening up a special department for the purpose.

Some years ago proposals were made to the Council of the Institute of Chemistry that they should re-issue the list, already out of print, taking the opportunity to revise some of the monographs in the light of experience, and to add to the number of them. This matter was considered fully and at some considerable length, and as a result the decision was made not to take any further steps; the view being that the action of the Institute of Chemistry in this matter was war emergency work, and not the proper function of the Institute as a professional body in ordinary circumstances.

We find, however, that there is a real need for such a work of reference. From our experience gained during

the past ten years we have made revisions in the monographs drawn up by the special Joint Committee, and we have evolved monographs for many other chemical substances, not only analytical reagents, but chemicals used in research, teaching, and, in fact, for what may comprehensively be termed "scientific purposes".

With a view to making the results of our experience generally available, we are now issuing in book form 158 of these monographs, which include those 88 substances for which standards were given in the official booklet.

Before taking this step we ascertained that the Councils of the Institute of Chemistry and the Society of Public Analysts would have no objection to our action, the object of which is that the work begun by the Joint Committee and extended byourselves should be available for the use and benefit of professional chemists generally.

THE BRITISH DRUG HOUSES LTD.

January, 1926.

## EXPLANATORY NOTES

Purpose of the This book defines, as exactly as possible, "A.R." Book the commercially attainable standards for many of the chemicals used for scientific purposes for which purity is of the first importance. Disapproval has often been expressed of the unsatisfactory nomenclature used in the chemical trade to distinguish different grades of purity. The most unsatisfactory description of all is that of "Chemically Pure" often abbreviated to "C.P." Chemically Pure implies absolute purity-a condition very desirable but never attained-and the practice of applying this term to any available grade of chemical is quite indefensible. It is the object of this book to give a precise meaning to the term "A.R." and to provide a clear definition of the purity of chemicals described as "A.R." and sold under the B.D.H. label.

Improved Monographs New methods for the detection of minute traces of many impurities have been evolved and advantage has been taken of these to render the "A.R." tests more delicate. The use of reagents for new purposes has in some cases revealed the presence of unsuspected impurities, and it has been necessary to devise new methods of purification and new tests for the detection of these impurities.

Additional Monographs A great amount of research work has been carried out in recent years in connection with general chemical testing and particularly with regard to biochemistry and the science of nutrition. This work has necessitated the application of tests of increasing delicacy for the detection and determination of the constituents of body fluids and of food

substances. For these tests substances of reagent purity are necessary, and the following may be cited: antimony trichloride, for the determination of the vitamin A (chromogen) in cod-liver oil; dimethyl-amino-benzaldehyde, for the colorimetric determination of the alkaloids of ergot of rye; picric acid, for the colorimetric determination of creatinine in urine.

Types of Wherever possible, methods have been used Tests for determining the actual amount of the impurity present; thus, arsenic limits are given for a number of chemicals used for arsenic testing, and full details are given of the methods employed in carrying out these determinations.

Most tests, however, do not lend themselves to quantitative statements of results; for these, limit tests have been adopted, and the smallest quantity that can be detected under the conditions of the test has, in most cases, been adopted as the maximum limit of that particular impurity. In this connection, it is of extreme importance that the conditions of the test be adhered to, and nowhere is this more noticeable than in testing for the presence of the sulphate radicle. A turbidity which is just distinguishable in a volume of 50 e.c. of water acidified with I e.e. of hydrochloric acid is produced in 1 hour by 0.1 milligram of SO3; in the presence of 5 c.c. of hydrochloric acid, however, it requires 0.8 milligram of SO3 to produce the same turbidity. In the tests for sulphates, therefore, the amount of acid used has been reduced to the minimum.

Where no length of time is stated, a period of five minutes is allowed. In several cases, the tests have been made more stringent by introducing longer time limits. In the case of tests dependent upon the pH of a solution, it is intended that neutral CO<sub>2</sub> free water should be used.

Chemicals for standardising Volumetric

Solutions

• The following chemicals conforming to the "A.R." standards may be used for standardising volumetric solutions. Appropriate factors will be found in each of the monographs:—

Antimony potassium tartrate Arsenious oxide Barium thiosulphate Ferrous ammonium sulphate Guanidine earbonate Potassium dichromate Potassium hydrogen tartrate Potassium dihydrogen phosphate Sodium carbonate Sodium chloride

It should be observed that all chemicals are apt to absorb or lose moisture if exposed to the air, even by diffusion through corks. Great care should be exercised, therefore, in keeping the chemicals required for standardising purposes in the same condition as that in which they are supplied.

Chemicals for preparing Buffer Solutions The following chemicals conforming to "A.R." standards are suitable for preparing buffer solutions for the determination of hydrogen ion concentrations.

Acetic acid
Amino-acetic acid
Boric acid
Citric acid
Potassium chloride
Potassium dihydrogen phosphate
Potassium hydrogen phthalate
Sodium phosphate anhydrous

Maximum Limits Under the heading of each monograph of Impurities is given a statement of the maximum limits of impurities. This does not imply that the amounts stated are present, but that these amounts represent the maximum permissible limits. In the great majority of cases, the amounts of impurity present are considerably less than the maximum.

Quantitative As far as possible, quantitative assays are prescribed and standard percentages specified, but in a few instances of salts which contain water of crystallisation and are not permanent in air, only the method of assay is given, no definite standard being laid down.

The molecular weights given are calculated from the International Atomic Weights, 1931.

It has not been considered necessary to acknowledge the many sources of the tests described, distributed as they are through many scientific publications. All have been the subject of extended experience in the B.D.H. Analytical Laboratories; some were originated there, and others have been modified in the light of experience gained.

THE BRITISH DRUG HOUSES LTD.

# THE B.D.H. BOOK OF A.R. STANDARDS

# ACETIC ACID GLACIAL A.R.

 $CH_3COOH = 60.03$ 

#### Maximum Limits of Impurities

Matter non-volatile at 100°			0.0005	per	cent.
Chloride (Cl)			0.0003	per	cent.
Sulphate (SO <sub>3</sub> )			0.0003	per	cent.
			0.0001	per	cent.
Bromine absorbed (Br)			0.006	per	cent.
Formate, sulphite and	ot	her		•	
oxidisable matter calcul-					
НСООН			0.04	per	cent.

A clear, colourless liquid with a characteristic odour; miscible with water, alcohol, and many oils.

# Freezing Point

Not below 15.6°.

#### Residua

10 c.c. evaporated to dryness on a watch-glass should not leave any appreciable residue.

# Hydrochloric Acid

5 c.c. diluted with 45 c.c. of water should not show any opalescence on addition of 1 c.c. of nitric acid and 1 c.c. of silver nitrate solution.

# Sulphuric Acid

50 c.c. evaporated to dryness on a water-bath, and the residue dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding 1 c.c. of barium chloride solution and standing for 12 hours.

# Heavy Metals and Iron

10 c.c. diluted with 20 c.c. of water and rendered slightly alkaline with ammonia should not show any appreciable darkening on addition of a few drops of sodium sulphide solution.

(Continued overleaf)

#### Reducing Substances

5 c.c. diluted with 15 c.c. of water and allowed to stand for 15 minutes with 0.5 c.c. of N/10 KMnO, should retain a pink tint.

Bromine Absorption

Introduce 20 c.c. into a 50 c.c. graduated stoppered flask, add 25 c.c. of water and 5 cc. of approximately decinormal bromine solution in glacial acetic acid. Shake and adjust with water to exactly 50 c.c. Titrate immediately 10 c.c. of this with the addition of 5 c.c. of potassium iodide solution against N/50  $\rm Na_{\rm s} S_{\rm s} O_{\rm s}$  using starch as indicator. Keep the remainder in a dark place at 20° C. for one hour and then titrate a second 10 c.c. against N/50 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. The difference between the two titrations should not exceed  $0\cdot 15$  c.c.

#### Formate, Sulphite and other oxidisable matter

Mix 10 e.e. with 1 c.e. of N/10 K2Cr2O, and 10 c.c. of sulphuric acid, cool, and allow to stand for 30 minutes; dilute with 50 c.c. of water, cool, add 1 c.c. of potassium iodide solution and titrate the liberated iodine with N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

Not less than 0.6 c.c. of Na2S2O3 should be required.

Titrate 2-3 grams diluted with water against N/1 NaOH, using phenolphthalein as indicator.

1 e.c. N/1 NaOH = 0.06003 gram CH<sub>2</sub>COOH

Not less than 99.5 per cent, should be indicated.

# ACETIC ANHYDRIDE A.R.

 $(CH_3CO)_2O = 102 \cdot 05$ 

Maximum Limits of Impurities

Muttan manl 0				
Matter non-volatile at 100°			0.0025	per cent.
Chloride (Cl)			_	
Sulphote (S())	•			1
Sulphate (SO <sub>3</sub> )			0.001	per cent.
Heavy Metals and Iron			0.001	
Dhoophumus 1 (D)	•	•	0.001	per cent.
Phosphorus compounds (P)			0.001	Der cont

A clear, colourless liquid with a pungent odour; soluble in alcohol, ether, and many other organic solvents. Slowly soluble in water with formation of acetic acid.

# Specific Gravity

1.08.

#### OF A.R. STANDARDS

#### Boiling Point

About 138°.

#### Residue

20 c.c. evaporated to dryness on a sand-bath should not yield more than 0.5 milligram of residue.

#### Hydrochloric Acid

5 c.c. dissolved in 45 c.c. of water should not show any opalescence on addition of 1 c.c. of nitric acid and 1 c.c. of silver nitrate solution.

#### Sulphate

10 c.c. dissolved in 100 c.c. of water should not show any precipitate on adding barium chloride solution and standing for 12 hours.

#### Heavy Metals and Iron

1 c.c. dissolved in 20 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Phosphorus Compounds

5 c.c. boiled with 10 c.c. of water and 5 c.c. of nitric acid should not give any yellow precipitate on adding 10 c.c. of ammonium molybdate solution, and standing in a warm place for 2 hours.

#### Higher Homologues

2 c.c. dissolved in 20 c.c. of water and nearly neutralised with sodium hydroxide solution should not have any unpleasant odour.

#### Other Impurities

20 c.e. with 5 c.c. of glycerol boiled gently under a reflux condenser for one hour and the excess of anhydride evaporated off, should form a solution free from flocculent matter on cooling and mixing with 50 c.c. of dilute nitric acid and allowing to stand for 80 minutes.

#### Assay

Weigh a flask containing 50 c.c. of N/1 NaOH, add 2 c.c. of the acetic anhydride, cool and reweigh. Allow to stand until the anhydride is decomposed, and titrate the excess of alkali against N/1  $\rm H_2SO_4$ , using phenolphthalein as indicator.

(Calculate to e.e. of N/1 NaOH for 1 g. of anhydride = a)

Weigh out 2 c.c. of the anhydride in a closed flask, add 20 c.c. of dry benzene, cool in a freezing mixture, and add a cooled solution of 10 c.c. of dry aniline in 20 c.c. of dry benzene. Allow to warm up to laboratory temperature. Add 50 c.c. of water, shake well and titrate against N/1 NaOH, using phenolphthalein as indicator. (Calculate c.c. of N/1 NaOH for 1 g. = b)

Then (a - b) 10.2 = per cent. actual acetic anhydride.

Not less than 95 per cent, should be present,

# ACETONE & A.R.

# $CH_3COCH_3 = 58.05$

# Maximum Limits of Impurities

Matter non-volatile at 100° . . 0.002 per cent.

Water-insoluble matter . . nil

Reaction . . . neutral

Oxygen absorbed (O) . . . 0.00025 per cent.

A clear, colourless, inflammable liquid with a characteristic odour; miscible in all proportions with water.

The aqueous solution should be neutral to litmus paper.

#### Specific Gravity

0.797-0.798.

#### **Boiling Range**

Should distil completely between 56° and 58°.

#### Residue

 $50\,\mathrm{c.c.}$  evaporated to dryness on a water-bath should not leave more than 1 milligram of residue.

#### Reducing Substances

20 c.c. allowed to stand in a cool place for 15 minutes with 0·1 c.c. of  $\bar{N}/10~KMnO_4$  should retain a pink colour.

#### Acidity

 $10\,c.c.$  diluted with  $10\,c.c.$  of neutral distilled water should not require more than  $0{\cdot}1$  c.c. of N/10 NaOH to produce a pink colour with phenolphthalein.

# Alkalinity

 $10\,c.c.$  diluted with  $10\,c.c.$  of neutral distilled water should not require more than  $0\cdot 2\,c.c.$  of  $N/10~H_2SO_4$  to produce a red colour with methyl red.

#### OF A.R. STANDARDS

# ACETYL BROMIDE A.R.

 $CH_3COBr = 122 \cdot 94$ 

#### Maximum Limits of Impurities

Non-volatile matter . . . o or per cent. Phosphorus compounds . . no reaction Sulphate (SO<sub>3</sub>) . . . o o4 per cent.

A colourless or slightly yellow furning liquid, soluble in water forming a clear solution.

#### Residue

10 c.c. evaporated to dryness on a water-bath and ignited gently should not leave more than 1 milligram of residue.

#### Phosphorus

1 c.c. boiled for 1 minute with 1 c.c. of water and 2 c.c. of nitric acid should not give any yellow precipitate on addition of ammonium molybdate solution.

#### Sulphate

1 c.c. dissolved in 10 c.c. of water should not show any immediate precipitate on addition of barium chloride solution.

#### Assay

Weigh a stoppered flask containing 50 c.c. of N/1 NaOH; add about 2 c.c. of the sample, cool and reweigh. Titrate the excess of alkali against N/1  $H_2SO_4$ , using phenolphthalcin.

1 e.e. N/1 NaOH = 0.06147 gram CH<sub>3</sub>COBr

Not less than 100 per cent. should be indicated. Dilute the neutralised liquid with water to produce 250 c.c., and titrate 50 c.c. of this against N/10  ${\rm AgNO_3}$ .

1 e.e.  $N/10 \text{ AgNO}_3 \equiv 0.01229 \text{ gram } CH_3COBr$ 

Not less than 95 per cent, should be indicated.

The issued product contains small amounts of acetic and hydrobromic acids.

# ACETYL CHLORIDE .A.R.

 $CH_3COCl = 78 \cdot 48$ 

# Maximum Limits of Impurities

Non-volatile matter . . . o or per cent.

Phosphorus compounds . . no reaction

Sulphate (SO<sub>3</sub>) . . . o o 4 per cent.

A colourless or slightly yellow fuming liquid, soluble in water forming a clear solution.

#### **Boiling Point**

About 51°.

#### Residue

10 c.c. evaporated to dryness on a water-bath and ignited gently should not leave more than 1 milligram of residue.

#### Phosphorus

1 c.c. boiled for 1 minute with 1 c.c. of water and 2 c.c. of nitric acid should not give any yellow precipitate on addition of ammonium molybdate solution.

#### Sulphate

1 c.c. dissolved in 10 c.c. of water should not show any immediate precipitate on addition of barium chloride solution.

#### Assay

Weigh a stoppered flask containing 50 c.c. of N/1 NaOH, add about 1 c.c. of the sample, cool and reweigh. Titrate the excess of alkali against N/1  $\rm H_2SO_1$ , using phenolphthalein.

#### 1 e.e. N/1 NaOH $\equiv$ 0·03924 gram CH<sub>3</sub>COCl

Not less than 100 per cent. should be indicated.

Dilute the neutralised liquid with water to produce 250 c.c., and titrate 50 c.c. of this against N/10 AgNO<sub>3</sub>.

1 c.c. N/10 AgNO<sub>3</sub> = 0.007848 gram CH<sub>2</sub>COCl

Not less than 98 per cent. should be indicated.

The issued product contains small amounts of acetic and hydrochloric acids.

#### OF A.R. STANDARDS

# ALUMINIUM OXIDE A.R. (CALCINED)

 $Al_9O_3 = 101.94$ 

# Maximum Limits of Impurities

Loss on ignition			1.0	per	cent.
Chloride (Cl)			0.005	per	cent.
Sulphate (SO <sub>3</sub> )			0.01	per	cent.
Iron (Fe) .			0.01	per	cent.

A dull white powder, insoluble in water and in dilute acids; incompletely soluble in sodium hydroxide solution.

#### Loss on Ignition

I gram heated to redness should not lose more than 10 milligrams.

#### Chloride

1 gram boiled with I c.c. of nitric acid and 20 c.c. of water and filtered should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram boiled with 1 c.c. of hydrochloric acid and 20 c.c. of water and filtered should not show more than a faint turbidity on addition of barium chloride solution.

#### Iron

1 gram boiled with 1 c.c. of hydrochloric acid and 20 c.c. of water and filtered should not show more than a faint blue colour on cooling and adding potassium ferrocyanide solution.

# ALUMINIUM SULPHATE A.R.

 $Al_2(SO_4)_3.18H_2O = 666 \cdot 40$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.001	per	cent.
Alkalis .			1.0	per	cent.
Heavy Metals			0.001	per	cent.
Iron (Fe) .			0.01	per	cent.
Ammonia (NE	$[_3)$ .		0.03	per	cent.

Damp white crystals or powder, very soluble in water forming a clear solution.

(Continued overleaf)

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Alkalis

Dissolve 1 gram in water and precipitate with ammonium chloride and ammonia. Filter and evaporate the filtrate to dryness and ignite gently. Not more than 10 milligrams of residue should be left.

#### Heavy Metals

1 gram dissolved in 40 c.c. of water should not show more than a faint darkening on addition of 10 c.c. of hydrogen sulphide solution.

#### Iron

 $0.1~{
m gram}$  dissolved in 50 e.e. of water should not show more than a faint blue colour on addition of potassium ferrocyanide solution.

#### Ammonia

1 gram heated with 10 c.c. of sodium hydroxide solution should not give any odour of ammonia.

# AMINO-ACETIC ACID

(Glycine, Glycocoll)

# $NH_2.CH_2.COOH = 75.05$

# Maximum Limits of Impurities

	٠			0.02	per cent.	
Chloride (Cl)				0.003	per cent.	
Sulphate (SO <sub>3</sub> )				0.01	per cent.	
Heavy Metals an	d Iı	on		0.001	per cent.	
Ammonia (NH <sub>2</sub> )				0.001	per cent.	

A white crystalline powder, soluble in water, forming a clear colourless solution.

#### Ash

1 gram moistened with sulphuric acid and ignited should not leave more than 0.5 milligram of residue.

#### OF A.R. STANDARDS

#### Chloride

1 gram dissolved in 10 c.c. of water and acidified with 0.2 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 10 c.c. of water and acidified with 0.2 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

#### Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show more than a faint darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Ammonia

1 gram dissolved in 50 c.c. of water should not show more than a slight yellow colour on addition of 2 c.c. of Nessler's reagent.

#### Assav

Dissolve 0.3 gram in 10 c.c. of water and add a neutral mixture of 10 c.c. of formaldehyde solution and 100 c.c. of alcohol. Titrate against N/10 NaOH, using phenolphthalein as indicator.

1 c.c. N/10 NaOH  $\equiv 0.007505$  gram NH<sub>2</sub>.CH<sub>2</sub>.COOH

Not less than 98.5 per cent. should be indicated.

# AMMONIA SOLUTION A.R. (STRONG)

 $\mathrm{NH_3} = 17\!\cdot\!03$ 

# Maximum Limits of Impurities

Non-volatile matter		0.001	per	cent.
Chloride (Cl)		0.0003	per	cent.
Sulphate (SO <sub>3</sub> ) .	•	0.0005	per	cent.
Sulphide		no reaction	n	
Heavy Metals and Iron		no reactio	n	
Tarry matter		no reactio	n	
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .		0.000005	per	cent.
Oxygen absorbed (O)		0.006	per	cent.

A clear, colourless liquid with a strong pungent odour; containing about 35 per cent. of  $\mathrm{NH}_3$ .

Specific Gravity
About 0.880.

(Continued overleaf)

#### Residue

100 c.c. evaporated to dryness on a water-bath should not leave more than 1 milligram of residue.

#### Chloride

10 c.c. evaporated nearly to dryness, diluted with 20 c.c. of water, and acidified with nitric acid, should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

10 c.c. evaporated nearly to dryness, diluted with 20 c.c. of water and acidified with hydrochloric acid, should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

#### Sulphide

10 c.c. should show no darkening in colour on the addition of 2 drops of potassium plumbite solution.

#### Heavy Metals and Iron

50 c.c. should not darken in colour on addition of 5 drops of sodium sulphide solution.

#### Tarry Matter

5 c.c. treated with 10 c.c. of water and 6 grams of citric acid should have no tarry odour.

#### Reducing Substances

Dilute 6 c.c. of sulphuric acid with 30 c.c. of water, cool and add 10 c.c. of the strong ammonia solution. To the resulting solution, add 0·1 c.c. of N/10 KMnO $_4$  and heat in a water-bath for 5 minutes. The pink colour should remain.

#### Arsenic

Limit 0.05 part per million.

Evaporate 100 c.c. nearly to dryness, dilute with 50 c.c. of water, add 10 c.c. of stannated hydrochloric acid and test as described on page 189.

# AMMONIUM ACETATE A.R.

 $\mathrm{CH_3COONH_4} = 77 \cdot 06$ 

# Maximum Limits of Impurities

Ash		0.01	ner	cent.
Chloride (Cl) .		0.001		
Sulphate (SO <sub>3</sub> ) .		0.01		
Heavy Metals and		0.0002		
Reaction		neutral	•	

Colourless hygroscopic crystals with a faint odour; readily soluble in water and in alcohol.

#### Ach

 $10\ \mathrm{grams}$  should not leave more than 1 milligram of residue on ignition.

#### Chloride

3 grams dissolved in 50 c.c. of water and acidified with 4 c.c. of nitrie acid should not show more than a faint opalescence on addition of silver nitrate solution.

# Sulphate

3 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding barium chloride solution, and allowing to stand for 6 hours.

# Heavy Metals and Iron

5 grams dissolved in 50 c.c. of water should not show more than a faint darkening on addition of 1 c.c. of ammonia and 2 drops of sodium sulphide solution.

#### Neutrality

I gram dissolved in 10 c.c. of recently boiled water should show a yellow or green colour with B.D.H. Universal Indicator.

# AMMONIUM ALUM .... A.R.

 $AlNH_4(SO_4)_2.12H_2O = 453.31$ 

#### Maximum Limits of Impurities

Chloride (Cl)				0.003	per	cent.
Heavy Metals				0.001	per	cent.
Iron (Fe) .				0.001	per	cent.
Alkalis and Alka	line	Earths		0.1	per	cent.

Colourless crystals, soluble in water, forming a clear colourless solution,

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Heavy Metals

1 gram dissolved in 20 c.c. of water should not show more than a faint darkening on addition of 10 c.c of hydrogen sulphide water.

#### Iron

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show more than a faint blue colour on addition of 1 c.c. of potassium ferrocyanide solution.

# Alkalis and Alkaline Earths

Dissolve 5 grams in water, add excess of ammonia and filter: wash the precipitate with water and evaporate the united filtrate and washings to dryness and ignite gently. The residue should not weigh more than 5 milligrams.

# AMMONIUM BICARBONATE € A.R.

 $NH_4HCO_3 = 79 \cdot 05$ 

# Maximum Limits of Impurities

Ash				0.02 per cent.	
				0.0005 per cent.	
Sulphate (SO <sub>3</sub> )				0.002 per cent.	
Tarry matter				no reaction	
Heavy Metals at	nd I	ron		0.0002 per cent.	

A white powder, soluble in water, forming a clear colourless solution.

5 grams should not leave more than 1 milligram of residue on ignition.

#### Chloride

5 grams boiled with 50 c.c. of water until most of the ammonia is dissipated should not show any opalescence on acidifying with nitric acid and adding silver nitrate solution.

#### Sulphate

5 grams boiled with 50 c.e. of water until most of the ammonia is dissipated should not show more than a faint turbidity on acidifying with hydrochloric acid, adding barium chloride solution and allowing to stand for 6 hours.

#### Tarry Matter

5-grams treated with 15 c.c. of water and 5 grams of citric acid should have no tarry odour.

#### Heavy Metals and Iron

5 grams dissolved in 40 e.e. of water should not show more than a faint darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Assay

Dissolve 8 grams in 50 c.c. of N/1 H<sub>2</sub>SO<sub>4</sub> and 50 c.c. of water and titrate the excess of acid against N/1 NaOH, using bromo-cresol green as indicator.

1 e.e. 
$$N/1 H_2SO_4 \equiv 0.07905 \text{ gram } NH_4HCO_3$$

Not less than 99 per cent. nor more than 101 per cent. should be indicated.

# AMMONIUM CARBONATE

#### Maximum Limits of Impurities

Ash				0.01	per cent.
Chloride (Cl)				0.0005	per cent.
Sulphate (SO <sub>3</sub> )				0.002	per cent.
Phosphate (P <sub>2</sub> O				0.001	per cent.
Thiocyanate	•			no reacti	ion
Heavy Metals ar	nd Ir	on		0.0001	per cent.
Tarry matter				no reacti	ion
Arsenic (As <sub>2</sub> O <sub>3</sub> )				0.00001	per cent.

White translucent lumps, with a strong ammoniacal odour; soluble in water.

Consists of an approximately equimolecular mixture of ammonium bicarbonate NH<sub>4</sub>HCO<sub>3</sub> and ammonium carbamate NH<sub>2</sub>COONH<sub>4</sub>.

#### Asi

10 grams should not leave more than 1 milligram of residue on ignition.

#### Chloride

5 grams boiled with 50 c.c. of water until most of the ammonia is dissipated should not show any opalescence on acidifying with nitric acid and adding silver nitrate solution.

# Sulphate

5 grams boiled with 50 c.c. of water until most of the ammonia is dissipated should not show more than a faint turbidity on acidifying with hydrochloric acid, adding barium chloride solution and allowing to stand for 6 hours.

#### Phosphate

• Poil 5 grams with 10 c.c. of water until most of the ammonia is dissipated. Add 5 c.c. of dilute sulphuric acid, 25 c.c. of water, 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B. No blue or green colour should develop.

#### Thiocyanate

2 grams dissolved in 20 c.c. of water, and acidified with hydrochloric acid, should not show a pink colour on addition of 1 drop of ferric chloride solution.

# Heavy Metals and Iron

10 grams boiled with 50 c.c. of water until most of the ammonia is dissipated should not show any green or brown colour on addition of 1 drop of sodium sulphide solution.

#### Tarry Matter

5 grams treated with 15 c.c. of water and 6 grams of citric acid should have no tarry odour.

#### Arcenie

Limit 0.1 part per million.

Boil 20 grams with 50 e.e. of water until most of the ammonia is dissipated, add 15 e.e. of brominated hydrochloric acid followed by a few drops of stannous chloride solution and test the resulting solution as described on page 189.

#### Assav

Dissolve about 2 grams in 50 c.c. of N/1  $\rm H_2SO_4$  and 50 c.c. of water and titrate the excess of acid against N/1 NaOH using bromo-cresol-green as indicator.

Not less than 31 per cent. of  $\rm NH_3$  should be indicated, equivalent to 95·3 per cent. of  $\rm NH_4HCO_3.NH_2COONH_4.$ 

# AMMONIUM CHLORIDE A.R.

 $NH_4Cl = 53 \cdot 50$ 

# Maximum Limits of Impurities

			o·oi per cent.
Sulphate (SO <sub>3</sub> ) .			o·oi per cent.
Phosphate (P <sub>2</sub> O <sub>5</sub> )			0.001 per cent.
Thiocyanate (SCN)			0.002 per cent.
Heavy Metals and Iro	n		0.0005 per cent.
Tarry matter .			no reaction
Arsenic (As <sub>0</sub> O <sub>0</sub> ).	_		0:0002 per cent.

 $\Lambda$  white crystalline powder, readily soluble in water, forming a clear solution, which should be neutral or at most slightly acid to methyl red.

#### Ash

 $10~{\rm grams}$  should not leave more than 1 milligram of residue on gentle ignition.

#### Sulphate

5 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding barium chloride, and allowing to stand for 6 hours.

(Continued overleaf)

#### Phosphate

Dissolve 5 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B and allow to stand for five minutes. No blue or green colour should be produced.

#### Thiocyanate

5 grams dissolved in 50 c.c. of water and acidified with hydrochloric acid should not show a pink colour on addition of 1 drop of ferric chloride solution.

# Heavy Metals and Iron

5 grams dissolved in 50 c.c. of water should not show more than a faint darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Tarry Matter

2 grams moistened with 1 c.c. of nitric acid and dried in separcelain dish on a water-bath should leave a perfectly white residue.

#### Arsenie

Limit 2 parts per million.

Test as described on page 189, using 5 grams of the ammonium chloride and 10 c.c. of stannated hydrochloric acid.

# AMMONIUM FLUORIDE

 $NH_4F = 37 \cdot 04$  $NH_4HF_2 = 57 \cdot 05$ 

### Maximum Limits of Impurities

Ash		0.03	per cent	ľ.
Chloride (Cl)		0.01	per cent	t.
Sulphate (SO <sub>3</sub> )		0.03	per cent	t.
Silicofluoride		passes	test	
Heavy Metals and Iron	_	0.000	s per cen	t.

White crystals, consisting of a mixture of the normal and acid fluorides; soluble in water, forming a clear colourless solution.

5 grams should not leave more than 1 milligram of residue on ignition.

#### Chloride

2 grams dissolved in 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

2 grams dissolved in 50 c.c. of water and acidified with 6 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Silicofluoride

2 grams dissolved in 10 c.c. of water should not show more than a faint opalescence on addition of 5 c.c. of a 20 per cent. solution of potassium chloride and 10 c.c. of alcohol.

#### Heavy Metals and Iron

2 grams dissolved in 20 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

# AMMONIUM MOLYBDATE A.R.

 $(NH_4)_6Mo_7O_{24}$ .  $4H_2O = 1236 \cdot 3$ 

Maximum Limits of Impurities

Chloride (Cl)				0.005	per cent.
Sulphate (SO <sub>3</sub> )				0.05	per cent.
Phosphate (P2O5)	)			0.0005	per cent.
Heavy Metals and	l Iron	า		0.001	per cent.

White crystals or crystalline lumps, sometimes with a greenish or yellowish tint.

#### Chloride

2 grams dissolved in 20 c.c. of water and slightly acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

# Sulphate

5 grams dissolved in 50 c.c. of water and acidified with nitric acid should not show any turbidity on adding barium nitrate solution, and allowing to stand for 6 hours.

#### Phosphate

Dissolve 0.25 gram in 50 c.c. of warm N/1 sulphuric acid; cool and add 5 c.c. of phosphate reagent B. No blue colour should be produced in 5 minutes.

# Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water and 1 c.c. of ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Assay

Treated as described under molybdic acid not less than 81 per cent, of MoO<sub>3</sub> should be indicated.

# AMMONIUM NITRATE A.R.

# $NH_4NO_3 = 80.05$

# Maximum Limits of Impurities

Ash			0.01	per cent.	
Chloride (Cl) .			0.0005	per cent.	
Sulphate (SO <sub>3</sub> ) .			0.01	per cent.	
Phosphate (P <sub>2</sub> O <sub>5</sub> )			0.0005	per cent.	
Thiocyanate (SCN)			0.002	per cent.	
Nitrite (N <sub>2</sub> O <sub>3</sub> ) .			0.0002	per cent.	
Heavy Metals and Ir	on	_	0.0002	per cent.	

Colourless, transparent crystals or crystalline powder, readily soluble in water.

#### Asl

 $10\ \mathrm{grams}$  should not leave more than 1 milligram of residue on ignition.

#### Chloride

5 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding barium chloride solution and allowing to stand for 6 hours.

#### Phosphate

Dissolve 5 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B and allow to stand for five minutes. No blue or green colour should be produced.

#### Thiocyanate

"5 grams dissolved in 50 c.c. of water and acidified with hydrochloric acid should not show any pink colour on addition of 1 drop of ferric chloride solution.

#### Nitrite

1 gram dissolved in 50 c.c. of water should not show any orange colour on addition of 1 c.c. of sulphuric acid and 1 c.c. of m-phenylenediamine sulphate solution.

# Heavy Metals and Iron

5 grams dissolved in 50 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

# AMMONIUM OXALATE <€0. → A.R.

 $(COONH_4)_9.H_9O = 142 \cdot 10$ 

# Maximum Limits of Impurities

Ash			-	0.02 per cent.
Chloride (Cl)				0.005 per cent.
Sulphate (SO <sub>3</sub> )				o·oi per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )				0.003 per cent.
Heavy Metals ar	nd I	ron		o · ooı per cent.
Reaction .				 neutral *

Colourless crystals, soluble in water, forming a clear colourless solution, which should be neutral in reaction (greenish-yellow to B.D.H. Universal Indicator).

#### Ael

5 grams should not leave more than 1 milligram of residue on ignition.

# Chloride

1 gram dissolved in 25 c.c. of water and acidified with 2·5 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of hot water and acidified with hydrochloric acid should not show any turbidity on adding barium chloride solution, and allowing to stand for 6 hours.

#### Nitrat

0.5 gram dissolved in 15 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 15 c.c. of sulphuric acid.

#### Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Assay

Dissolve 0.3 gram in hot water, add dilute sulphuric acid, and titrate against N/10 KMnO<sub>4</sub> at a temperature of about 60°.

1 c.c.  $N/10 \text{ KMnO}_4 \equiv 0.007105 \text{ gram } (COONH_4)_2.H_2O$ 

Not less than 99 per cent. should be indicated.

# AMMONIUM PERSULPHATE

 $NH_4SO_4 = 114 \cdot 1$ 

# Maximum Limits of Impurities

Ash			0.1	per	cent.
Chloride (Cl)			0.001	per	cent.
Manganese (Mn)			0.0002	per	cent.

White granular crystals or crystalline powder, readily soluble in water, forming a clear solution.

#### Ash

 ${\bf 5}$  grams should not leave more than  ${\bf 5}$  milligrams of residue on ignition.

# Chloride

1 gram dissolved in 50 c.c. of water should not show any opalescence on addition of 0.2 c.c. of silver nitrate solution.

#### Manganese

Warm 5 grams with 20 c.c. of water, 1 c.c. of nitric acid and 1 c.c. of N/10  ${\rm AgNO_3}$  to 90° for 5 minutes and cool. No pink colour should be produced.

#### Assay

Mix about 0.4 gram with 25 c.c. of water, 3 grams of potassium iodide, and 10 c.c. of dilute sulphuric acid, allow to stand for 20 minutes and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c.  $N/10 \text{ Na}_2S_2O_3 \equiv 0.01141 \text{ gram } NH_4SO_4$ 

Not less than 97 per cent. should be indicated.

# AMMONIUM PHOSPHATE A.R.

(Dibasic)

 $(NH_4)_2HPO_4 = 132 \cdot 11$ 

# Maximum Limits of Impurities

Chloride (Cl) .				0.003	per cent.
Sulphate (SO <sub>2</sub> ) .					per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> ) .					per cent.
Heavy Metals and	Iron				per cent.
Alkalis and other	Metals				per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ).					per cent.
(1402 0 3)	•	•	•	0 0001	per centi

Colourless crystals, readily soluble in water.

#### Chloride

2 grams dissolved in 20 c.c. of water and acidified with nitric acid should not show more than a slight opalescence on addition of silver nitrate solution.

### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with hydrochloric acid should not show any turbidity or precipitate on adding barium chloride solution, and allowing to stand for 6 hours.

#### Nitrate

2 grams dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Heavy Metals and Iron

2 grams dissolved in 50 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

# Alkalis and Other Metals

Dissolve 2 grams in 100 c.c. of water, precipitate the phosphate by the addition of a slight excess of lead acetate and filter; remove the lead from the filtrate by means of hydrogen sulphide, filter, evaporate the filtrate to dryness and ignite. The residue should not exceed 3 milligrams.

(Note.—An allowance must be made for any alkalis in the lead acetate.)

#### Arsenic

Limit 1 part per million.

Test as described on page 189, using 5 grams of the ammonium phosphate and 12 c.c. of stannated hydrochloric acid.

#### Assay

Dissolve 5 grams in water, and titrate against N/1  $\rm H_2SO_4$  using bromo-phenol blue as indicator.

1 e.e. 
$$N/1 H_2SO_4 \equiv 0.1321 \text{ gram } (NH_4)_2HPO_4$$

Not less than 97 per cent, should be indicated.

# AMMONIUM SULPHATE

 $(NH_4)_0SO_4 = 132 \cdot 14$ 

# Maximum Limits of Impurities

Ash						0.02	per	cent.
Moist	ıre					0.4	per	cent.
Chlori	ide (C	(1)				0.001	per	cent.
Phosp	hate (	P,O,	)			0.001	per	cent.
Thioc	yanate	(SC	N)			0.006	per	cent.
Heavy	Meta	ıls`ano	Iror	1		0.0005	per	cent.
Arseni	c (As	$(aO_a)$	_			1000.0	per	cent.

A white crystalline powder, readily soluble in water, forming a clear, colourless solution, which should be neutral or not more than faintly acid to methyl red.

5 grams should not leave more than 1 milligram of residue on ignition.

#### Moisture

5 grams dried at 100° should not lose more than 20 milligrams in weight.

# Chloride

2 grams dissolved in 20 c.c. of water, and acidified with 1 c.c. of nitric acid, should not show more than a faint opalescence on addition of silver nitrate solution.

Dissolve 5 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B and allow to stand for 5 minutes. No blue or green colour should be produced.

### \*Thiocyanate

Phosphate

2 grams dissolved in 10 c.c. of water, and acidified with 1 c.c. of hydrochloric acid, should not show a pink colour on addition of 1 drop of ferrie chloride solution.

2 grams dissolved in 50 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Arsenic

Limit 1 part per million.

Heavy Metals and Iron

Test described on page 189, using 5 grams of the ammonium sulphate and 10 c.c. of stannated hydrochloric acid.

# AMMONIUM SULPHIDE SOLUTION A.R.

# Maximum Limits of Impurities

Non-volatile residue			0.01	per	cent.
Carbonate (CO <sub>2</sub> )	_		0.005	per	cent.

An ammoniaeal solution of ammonium sulphide containing more or less polysulphide. Colourless when freshly prepared, but gradually becoming yellow with formation of polysulphide on keeping.

### Residue

 $10~\mathrm{c.c.}$  evaporated to dryness and ignited gently should not leave more than 1 milligram of residue.

# Carbonate and Sulphate

10 c.c. should not give any precipitate on adding 3 c.c. of calcium chloride solution, and allowing to stand for 1 hour.

# Assay

Add 5 c.c. together with 10 c.c. of ammonia to a solution of 3 grams of copper sulphate (CuSO<sub>4</sub>.5H<sub>2</sub>O) dissolved in 30 c.a. of water and filter. The filtrate should not be coloured blue, showing presence of at least 8·19 per cent. of hydrogen sulphide.

# AMMONIUM THIOCYANATE ... A.R.

# $NH_aSCN = 76 \cdot 11$

# Maximum Limits of Impurities

				0.02	per	cent.
Chloride (Cl)				0.005	per	cent.
Sulphate (SO <sub>3</sub> )				0.01	per	cent.
Isothiocyanate				no reac	tion	
Heavy Metals ar	ıd I	ron		0.0005	per	cent.

Colourless deliquescent crystals, readily soluble in water and in alcohol.

#### Ash

10 grams should not leave more than 2 milligrams of residue on ignition.

#### Chloride

Dissolve 1 gram in 30 c.c. of hydrogen peroxide (20 volumes), add 1 gram of sodium hydroxide and rotate the flask gently from time to time until a vigorous reaction commences. When this has abated, add a further 30 c.c. of hydrogen peroxide and boil for 2 minutes; cool, acidify with nitric acid and add 1 c.c. of silver nitrate solution. Not more than a faint opalescence should be produced.

# Sulphate

5 grams dissolved in 50 c.c. of water and acidified with hydrochloric acid should not show any turbidity on adding barium chloride solution, and allowing to stand for 1 hour.

### Isofhiocyanate

5 grams dissolved in 25 c.c. of water, 3 c.c. of silver nitrate solution added and the precipitate redissolved by thorough shaking, should not darken in colour on warming with 20 c.c. of ammonia for 15 minutes.

# Heavy Metals and Iron

5 grams dissolved in 50 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should form a colourless solution, which should not darken on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

# AMMONIUM VANADATE A.R.

 $NH_4VO_3 = 116.99$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.003	per	cent.
Sulphate (SO <sub>3</sub> )			0.01	per	cent.

A white crystalline powder, readily soluble in water, forming a clear colourless solution.

#### Chloride

Dissolve 1 gram in 50 c.c. of warm water, saturate with sulphur dioxide, acidity with 3 c.c. of sulphuric acid and add a few drops of silver nitrate solution. Not more than a faint opalescence should be produced.

# Sulphate

Dissolve 1 gram in 50 c.c. of warm water, acidify with 1 c.c. of hydrochloric acid, add 1·5 gram of hydroxylamine hydrochloride, warm to 60° for 3 minutes and add 1 c.c. of barium chloride solution. No turbidity should be produced.

### Assay

Dissolve 0.5 gram in 30 c.c. of water, add 5 c.c. of dilute sulphuric acid, warm on a water-bath and pass in sulphur dioxide until reduction is complete and the solution is bright blue. Remove the excess of  $SO_2$  by boiling gently and passing in a stream of  $CO_2$ ; cool and titrate with N/10 KMnO<sub>4</sub>.

1 c.c. N/10 KMnO<sub>4</sub> = 0.0117 gram NH<sub>4</sub>VO<sub>3</sub>

Not less than 98.5 per cent, should be indicated.

# AMYL ACETATE A.R.

# $CH_3COOC_5H_{11} = 130 \cdot 11$

# Maximum Limits of Impurities

Non-volatile matter . . o o i per cent.

Water . . . absent

Free Acid (CH<sub>3</sub>COOH) . 0.06 per cent.

A colourless or very pale yellow liquid with a characteristic odour. Insoluble in water; miscible with alcohol, ether and other organic solvents.

### Specific Gravity

0.874 to 0.878.

#### **Boiling Range**

Not less than 90 per cent. should distil between  $136^{\circ}$  and  $142^{\circ}$ .

#### Residue

 $10\ \mathrm{c.c.}$  evaporated on a water-bath should not leave more than 1 milligram of residue.

#### Solubility

5 c.c. should form a clear mixture with 5 c.c. of benzene. 5 c.c. should form a clear mixture with 5 c.c. of carbon disulphide. 1 c.c. should dissolve to a clear solution in 20 c.c. of 45 per cent. alcohol.

## Free Acid

•1 •gram dissolved in 10 c.c. of alcohol should not require more than 0·1 c.c. of N/10 KOH (alcoholic) to produce a pink colour with phenolphthalein.

### Assay

Saponify 2 to 3 grams dissolved in 25 c.c. of alcohol with 25 c.c. of N/1 KOH (alcoholic) for 1 hour. Titrate the excess of alkali against N/1  $\rm H_2SO_4$ , using phenolphthalein as indicator.

1 c.c. N/1 KOH = 0.1301 gram CH<sub>3</sub>COOC<sub>5</sub>H<sub>11</sub>

Not less than 97.5 per cent, should be indicated.

# AMYL ALCOHOL A.R.

# $C_5H_{11}OH = 88 \cdot 10$

# Maximum Limits of Impurities

Non-volatile matter . . 0.001 per cent. Colour with H2SO4. . not more than light brown . no reaction

Oily impurities

 ${\bf A}$  clear, colourless or very pale yellow liquid with a characteristic odour. Consists principally of iso-amyl alcohol.

Specific Gravity 0.812 to 0.815.

Boiling Range

130° to 132°.

10 c.c. evaporated to dryness on a water-bath should not leave any residue.

# Furfural and Organic Impurities

5 c.c. shaken with 5 c.c. of sulphuric acid should not produce more than a yellow or light brown colour.

# Solubility

10~c.c. should dissolve completely in 10~c.c. of hydrochloric acid (sp. gr.  $1\cdot17)$  and the addition of  $1\cdot5~c.c.$  of water should cause a

#### Oily Impurities

2 c.c. dissolved in a cold mixture of 10 c.c. of sulphuric acid and 10 c.c. of water and centrifuged in a Gerber milk tube should not show any oily layer or globules.

# AMYL ALCOHOL

(Pyridine and Nitrogen Free)

### Maximum Limits of Impurities

This should pass the following test in addition to those in the preceding monograph:—

Shake 50 c.c. with 20 c.c. of 1 per cent. sulphuric acid: separate the acid layer and repeat the shaking with a second 20 c.c. of the acid. Mix the acid liquids, make alkaline with sodium hydroxide and distil. Collect the distillate in 10 c.c. of N/10 H<sub>2</sub>SO<sub>4</sub> and titrate the excess of acid against N/10 NaOH, using methyl orange or bromo-phenol blue as indicator.

Not less than 9.75 c.c. of N/10 NaOH should be required.

# AMYL ALCOHOL & A.R.

(For Milk Testing)

Maximum Limits of Impurities

Colour with  $H_2SO_4$  . not more than light brown Oily impurities . no reaction

# Boiling Range

125° to 132°.

This should pass the tests in the monograph on Amyl Alcohol A.R. for :—

Furfural and Organic Impurities

Solubility

Oily Impurities

# AMYL NITRITE ... A.R.

 $C_5H_{11}ONO = 117 \cdot 10$ 

A clear, pale yellow, mobile liquid with a characteristic odour; insoluble in water, but miscible with alcohol.

Specific Gravity •0.870 to 0.875.

Boiling Range

90 per cent. should distil between 90° and 100°.

Assay

Dilute 5 c.c. with sufficient alcohol to produce 100 c.c. Introduce 5 c.c. of this (= 0.25 c.c. of amyl nitrite) into a brine-charged nitrometer, add 5 c.c. of potassium iodide solution and 5 c.c. of dilute sulphuric acid, shake well during 5 minutes, allow to stand for 15 minutes, and measure the liberated nitric oxide. Correct for temperature and pressure.

1 c.c. NO at 0° C. and 760 mm.  $\equiv$  0.0052 gram  $C_5H_{11}ONO$ 

Not less than 90 per cent. by weight should be indicated. Note.—This is approximate only, as no correction is made for tension of aqueous and alcoholic vapour.

# ANILINE A.R.

 $C_6H_5NH_2 = 93.06$ 

# Maximum Limits of Impurities

Non-volatile matter . . . . 0 · 005 per cent. Nitrobenzene . . . . no reaction

An almost colourless oily liquid when freshly distilled, but which rapidly darkens to a reddish-brown colour: slightly soluble in water.

Specific Gravity
About 1.027.

(Continued overleaf)

### Freezing Point About - 7°.

Boiling Range 182° to 184°.

#### Solubility

5 c.c. should dissolve in 30 c.c. of dilute hydrochloric acid to clear solution free from any odour of nitrobenzene.

# Non-volatile Matter

10 c.c. on evaporation and gentle ignition should not leave more than 0.5 milligram of residue.

# ANTIMONY POTASSIUM TARTRATE A.R.

 $\mathrm{KSbO.C_4H_4O_6} = 324 \cdot 9$ 

Maximum Limits of Impurities

Moisture . . . . . . o · 1 per cent.

A white or pale cream coloured crystalline powder, soluble in water, forming a clear solution.

#### Moisture

 $_{\, 5}$  grams should not lose more than 5 milligrams on drying at  $100^{\circ\, 6} {\rm for} \ 1 \ {\rm hour.}$ 

#### Assay

Dissolve 0.8 gram in 50 c.c. of water, add 5 grams of sodium potassium tartrate and 1 gram of sodium bicarbonate and titrate against N/10 I.

1 c.c. N/10  $I \equiv 0.016245$  gram KSbO .  $C_4H_4O_6$ 

Not less than 99.9 per cent. should be indicated.

# ANTIMONY TRICHLORIDE A.R.

 $SbCl_3 = 228 \cdot 13$ 

# Maximum Limits of Impurities

Oxychloride			trace
Iron (Fe) .			o · oooi per cent.

Colourless crystals, fuming in moist air; soluble in water acidified with hydrochloric acid; soluble in chloroform forming a solution which should not be more than faintly turbid.

#### Inc.

1 gram boiled with 1 c.c. of nitric acid and 10 c.c. of water and cooled should not show more than a slight pink coloration on addition of 1 c.c. of ammonium thiocyanate solution.

#### Assay

Dissolve 0.5 gram in 30 c.c. of water containing in solution 4 grams of sodium potassium tartrate, add 2 grams of sodium bicarbonate and titrate with  $N/10\ I.$ 

1 c.c. N/10 I = 0.01141 gram SbCl<sub>3</sub>

Not less than 99 per cent. should be indicated.

# ARSENIOUS OXIDE A.R.

 ${\rm As_2O_3} = 197 \cdot 86$  Maximum Limits of Impurities

A dense white powder.

#### Solubility, Heavy Metals and Iron

1 gram should dissolve in 20 c.c. of ammonia, forming a clear colourless solution, and should not give a precipitate or more than a faint darkening on addition of hydrogen sulphide solution.

(Continued overleaf)

#### Residue

2 grams should not leave more than 0.5 milligram of residue on ignition.

#### Sulphide

5 grams dissolved in sodium hydroxide solution should not show any darkening on addition of 1 drop of lead acetate solution.

# Antimony, Tin and Cadmium

The precipitate produced by dissolving 0.5 gram in 50 c.c. of water and 1 c.c. of hydrochloric acid and treating with hydrogen sulphide should, after washing, be completely soluble in ammonium carbonate solution.

#### Assay

Dissolve  $0\cdot 2$  gram in dilute sodium hydroxide solution, render faintly acid with hydrochloric acid, add about 2 grams of sodium bicarbonate and titrate against N/10 I.

1 c.c. N/10 
$$I \equiv 0.004947$$
 gram  $As_2O_3$ 

Not less than 99.8 per cent, of As<sub>2</sub>O<sub>3</sub> should be indicated.

# BARIUM ACETATE

 $(CH_3COO)_2Ba$ .  $H_2O = 273 \cdot 42$ 

# Maximum Limits of Impurities

Chloride (Cl)		0.003 per cent.
Nitrate (N2O5)		0.002 per cent.
Heavy Metals and Iron		0.001 per cent.
Alkalis and other Metals		O·I per cent.

White crystals or crystalline powder, readily soluble in water, forming a clear solution.

#### Chloride

1 gram dissolved in 50 c.c. of water should not produce more than a faint opalescence on addition of 1 c.c. of nitric acid and 1 c.c. of silver nitrate solution.

#### Nitrate

I gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Heavy Metals and Iron

I gram dissolved in 10 c.c. of water and acidified with 1 c.c. of acetic acid should not show more than a faint darkening on addition of excess of ammonia and 1 drop of sodium sulphide solution.

### Alkalis and other Metals

Dissolve 5 grams in 200 c.c. of water, add 25 c.c. of dilute sulphuric acid and boil gently; allow to stand for two hours and filter. The filtrate evaporated to dryness and ignited should not leave more than 5 milligrams of residue.

# BARIUM CARBONATE A.R.

 $\mathrm{BaCO_3} = 197 \!\cdot\! 36$ 

# Maximum Limits of Impurities

Chloride (Cl) .				0.001	per	cent.
Sulphate and Acid-insol	uble:	matte	r	nil	-	
Nitrate (N <sub>2</sub> O <sub>5</sub> ) .				0.003	per	cent.
Heavy Metals .				0.001	per	cent.
Iron (Fe)				0.002	per	cent.
Alkalis and other Meta	ls			O·I	per	cent.

A white powder or friable masses, insoluble in water.

#### Chloride

1 gram dissolved in 20 c.c. of water and 3 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphat

5 grams should dissolve in 100 c.c. of dilute hydrochloric acid, forming a clear, colourless solution.

#### Nitrate

1 gram dissolved in 10 c.c. of acetic acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid

#### Heavy Metals

1 gram dissolved in 25 c.c. of water and 5 c.c. of acetic acid should not show more than a faint darkening on addition of 20 c.c. of hydrogen sulphide water.

(Continued overleaf)

#### Inn

0.5 gram dissolved in 50 c.c. of water and 2 c.c. of hydrochloric acid should not show more than a faint blue colour on addition of 1 c.c. of potassium ferrocyanide solution.

# Alkalis and other Metals

Dissolve 5 grams in 10 c.c. of hydrochloric acid and 200 c.c. of water, add 25 c.c. of dilute sulphuric acid, boil gently to granulate the precipitate, allow to stand for two hours and filter. The filtrate evaporated to dryness and ignited should not leave more than 5 milligrams of residue.

#### Assay

Dissolve 4 grams in 50 c.c. of N/1 HCl and 50 c.c. of water and titrate the excess of acid against N/1 NaOH, using bromophenol blue as indicator.

1 c.c. N/1 HCl = 0.09868 gram BaCO<sub>3</sub>

Not less than 99.0 per cent. should be indicated.

# BARIUM CHLORIDE A.R.

 $\mathrm{BaCl_2}.\,2\mathrm{H_2O} = 244\cdot30$ 

Maximum Limits of Impurities

Nitrate $(N_2O_5)$		0.002	per cent.
Heavy Metals and Iron		0.0002	per cent.
Alkalis and other Metals		0.1	per cent.
Calcium and Strontium		0.2	per cent.

Colourless crystals, readily soluble in water, forming a clear colourless solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Heavy Metals and Iron

5 grams dissolved in 50 c.c. of water should not show more than a faint darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Alkalis and other Metals

Dissolve 5 grams in 200 c.c. of water, add 25 c.c. of dilute sulphuric acid, and boil gently; allow to stand for two hours, and filter. The filtrate evaporated to dryness and ignited should not leave more than 5 milligrams of residue.

### Calcium and Strontium

Shake 1 gram in fine powder with 20 c.c. of absolute ethyl alcohol for 5 minutes and filter; the filtrate evaporated to dryness should not eave more than 2 milligrams of residue.

# BARIUM HYDROXIDE

 $Ba(OH)_{2} \cdot 8H_{2}O = 315 \cdot 50$ 

### Maximum Limits of Impurities

Chloride (Cl)				0.003 per cent.
Sulphate .				nil .
Nitrate (N <sub>2</sub> O <sub>5</sub> )		,		0.002 per cent.
Carbonate .	,			trace
Sulphide .				no reaction
Heavy Metals				o ooi per cent.
Alkalis and other	Meta			0.2 per cent.

#### Colourless crystals.

#### Chloride

1 gram dissolved in 10 c.c. of water and 2 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 2 c.c. of hydrochloric acid and 10 c.c. of water should form a clear solution.

#### Nitrate

1 gram dissolved in 10 c.c. of acetic acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Carhonate

1 gram should dissolve in 50 c.c. of  $\mathrm{CO}_2\text{-free}$  water with only a slight milkiness.

# Sulphide

1 gram dissolved in 50 c.c. of water should not darken in colour on addition of 1 drop of lead acetate solution.

(Continued overleaf)

#### Heavy Metals

1 gram dissolved in 10 c.c. of acetic acid and 10 c.c. of water should not show any darkening on addition of 20 c.c. of hydrogen sulphide water.

# Alkalis and other Metals

Dissolve 5 grams in 100 c.c. of water and 5 c.c. of hydrochloric acid, add 25 c.c. of dilute sulphuric acid, boil gently, allow to stand for two hours and filter. The filtrate evaporated to dryness and ignited should not leave more than 10 milligrams of residue.

#### Assay

Dissolve about 5 grams in water and titrate against N/1 HCl using methyl red as indicator.

1 e.c. N/1 HCl  $\equiv$  0 · 1578 gram Ba(OH)<sub>2</sub> · 8H<sub>2</sub>O

Not less than 99 per cent, should be indicated.

# BARIUM NITRATE

 $Ba(NO_3)_9 = 261 \cdot 38$ 

Maximum Limits of Impurities

 Moisture
 .
 .
 .
 0 · 2
 per cent.

 Chloride (Cl)
 .
 .
 .
 0 · 001
 per cent.

 Heavy Metals and Iron
 .
 .
 .
 0 · 001
 per cent.

 Alkalis and other Metals
 .
 .
 .
 .
 .
 .
 .

Colourless crystals, soluble in water forming a clear solution, neutral to litmus.

#### Moisture

5 grams dried at 100° should not lose more than 10 milligrams in weight.

#### Chloride

I gram dissolved in 25 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

# Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

# Alkalis and other Metals

Dissolve 5 grams in 100 c.c. of water, add 30 c.c. of dilute sulphuric acid, boil gently, allow to stand for two hours and filter. The filtrate, evaporated to dryness and ignited, should not leave more than 5 milligrams of residue.

# BARIUM THIOSULPHATE

 $BaS_0O_3$ .  $H_0O = 267.5$ 

A white crystalline powder, slightly soluble in water.

### Assay

Titrate 1.3376 grams suspended in 100 c.c. of water in a stoppered flask against  $N/10\,$  I.

(Owing to the slight solubility of this salt the reaction is very slow, and vigorous shaking is required.)

1 c.c. N/10  $I \equiv 0.02675$  gram  $BaS_2O_3$ .  $H_2O$ 

Not less than 99.8 per cent. should be indicated.

# BENZENE A.R.

 $C_6H_6=78\cdot05$ 

Maximum Limits of Impurities

Non-volatile matter . . . o · oor per cent.
Thiophen . . . no reaction
Other Sulphur compounds . o · ooo5 per cent.

A clear, colourless liquid with a characteristic odour. Insoluble in water; miscible with alcohol and ether.

# Specific Gravity

About 0.884.

# Freezing Point

5° to 6°.

# Boiling Point

80° to 81°.

# Residue

10 c.c. evaporated on a water-bath should not leave any residue.

#### Thiophen and Organic Impurities

Shake 50 c.c. with 20 c.c. of sulphuric acid. the acid layer may become yellow, but should not be coloured brown, and on addition of a trace of isatin, and again shaking, a blue or green colour should not be produced.

### Sulphur Compounds

Boil 10 c.c. with 1 c.c. of absolute alcohol and 3 c.c. of potassium plumbite solution for 15 minutes under a reflux condenser, and allow to stand for 5 minutes. The aqueous layer should remain colourless.

# BENZIDINE A.R.

 $(C_6H_4NH_9)_9 = 184 \cdot 11$ 

# Maximum Limits of Impurities

Ash .				0.1	per cent.
Moisture				1.0	per cent.
Insoluble i	nil	_			
Sulphate (	SO <sub>3</sub> )			0.01	per cent.

A pale buff coloured powder, completely soluble in alcohol and in dilute hydrochloric acid.

# Melting Point

128° to 129°.

#### Ash

5 grams moistened with sulphuric acid and ignited should not leave more than 5 milligrams of residue.

#### Moisture

 $2~{\rm grams}$  dried at  $110^{\circ}$  should not lose more than  $20~{\rm milligrams}$  in weight.

# Sulphate

1 gram dissolved in 30 c.c. of N/1 HCl should not show more than a faint turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 2 hours.

# BENZOIC ACID

 $C_6H_5COOH = 122 \cdot 05$ 

Calorific value per gram weighed in air 6324 calories

# Maximum Limits of Impurities

Ash		0.03	per cent.
Chlorine compounds (Cl)		0.02	per cent.
Heavy Metals and Iron		0.0002	per cent.
Oxygen absorbed (O)		0.02	per cent.

White needle crystals, readily soluble in alcohol and in ether, slightly soluble in water.

# Melting Point

121° to 122°.

#### Ash

5 grams should not leave more than 1 milligram of residue on ignition.

# Chlorine Compounds

Mix 5 grams with 3 grams of anhydrous sodium carbonate, transfer to 3 small porcelain crucible and fill the latter completely with more sodium carbonate well pressed down. Place a small platinum dish upside down over the crucible and invert the whole quickly. Add more sodium carbonate until the inverted crucible is half buried and heat strongly for 30 minutes. Cool and dissolve the mass in dilute nitric acid, add 5 c.c. of N/10 AgNO<sub>3</sub> and titrate the excess of silver against N/10 NH<sub>4</sub>SCN.

1 c.c.  $N/10 \text{ AgNO}_3 \equiv 0.0035457 \text{ gram Cl}$ 

# Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water and 5 c.c. of ammonia should produce a clear colourless solution, and should not show more than the slightest darkening on addition of one drop of sodium sulphide solution.

#### Reducing Substances

1 gram dissolved in 100 c.c. of hot water and acidified with 10 c.c. of sulphuric acid should not require more than 0.25 c.c. of N/10 KMnO<sub>4</sub> to produce a pink colour.

#### Assay

Dissolve about 2 grams in 10 c.c. of alcohol, add 30 c.c. of water, and titrate against N/1 NaOH, using phenol red as indicator.

1 c.c. N/1 NaOH  $\equiv$  0 · 12205 gram C<sub>6</sub>H<sub>5</sub>COOH

Not less than 99.8 per cent. should be indicated.

# BENZOYL CHLORIDE A.R.

 $\mathrm{C_6H_5COCl} = 140 \cdot 5$ 

# Maximum Limits of Impurities

Non-volatile matter . . . o · ot per cent. Iron (Fe) . . . . o · ool per cent. Phosphorus compounds . . no reaction Sulphur compounds . . no reaction

An almost colourless liquid, fuming in moist air. Slowly but completely soluble in sodium hydroxide solution.

#### Non-volatile Matter

 $10~{\rm grams}$  evaporated and gently ignited should not leave more than 1 milligram of residue.

#### Iron

1 gram shaken with 50 c.c. of water should not show a blue colour with potassium ferrocyanide solution.

# Phosphorus Compounds

1 gram boiled for 1 minute with 1 c.c. of water and 2 c.c. of nitric acid should not give any yellow precipitate on addition of ammonium molybdate solution.

# Sulphur Compounds

1 gram boiled for 1 minute with 1 c.c. of water and 2 c.c. of nitric acid and diluted with 20 c.c. of water should not show any turbidity on addition of 1 c.c. of barium chloride solution.

# BORIC ACID

# $H_3BO_3=61\cdot 84$

# Maximum Limits of Impurities

Chloride (Cl)		0.001	per cent.
Sulphate (SO <sub>3</sub> )		0.01	per cent.
Heavy Metals and Iron		0.0005	per cent.
Calcium (Ca)		0.002	per cent.
Arsenic (As-O-)		0.0005	ner cent

White crystalline flakes, slightly soluble in water, forming a clear colourless solution.

#### Chloride

2 grams dissolved in 50 c.c. of hot water should not show more than a faint opalescence on addition of silver nitrate solution and 1 c.c. of nitric acid.

# Sulphate

2 grams dissolved in 50 c.c. of hot water should not show any turbidity on adding barium chloride solution and 1 c.c. of hydrochloric acid and allowing to stand for 15 minutes,

### Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water and 5 c.c. of ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Calcium

5 grams dissolved in 100 c.c. of water and 10 c.c. of ammonia should not show any turbidity on adding ammonium oxalate solution and allowing to stand for 1 hour.

#### Arsenic

Limit 5 parts per million.

Dissolve 2 grams with 5 grams of citric acid in 50 c.c. of hot water, add 10 c.c. of stannated hydrochloric acid and test as described on page 189.

### Assay

Titrate 3.092 grams dissolved in a mixture of 50 c.c. of glycerol and 50 c.c. of water against  $N/1\,$  NaOH, using phenolphthalein as indicator.

1 c.c. N/1 NaOH = 
$$0.06184$$
 gram  $H_3BO_3$ 

Not less than 99.5 per cent. should be indicated.

# BROMINE A.R.

# $Br = 79 \cdot 916$

# Maximum Limits of Impurities

Non-volatile residue		0.01	per cent.	
Iodine (I)		0.1	per cent.	
Chlorine (Cl) .		0.1	per cent.	
Sulphate (SO <sub>3</sub> ) .		0.005	per cent.	
Organic compounds		no rea	ction	
Arsenic $(As_2O_3)$ .		0.000	per cent.	
			-	

A dark reddish-brown fuming liquid, slightly soluble in water.

### Specific Gravity

About 3.18.

#### Residue

3 c.c. evaporated in a porcelain dish on a water-bath should not leave more than 1 milligram of residue.

#### Indine

#### lodin

Shake 1 c.c. with 50 c.c of water and 2 grams of granulated zine until the bromine is reduced, and filter. The filtrate should not show a blue colour on addition of starch mucilage and 0·1 c.c. of ferric chloride solution.

#### Chlorine

Shake 3 c.c. with 0.5 gram of zinc dust and 5 c.c. of water for 10 minutes and evaporate to dryness on a water-bath. Dissolve the residue in 75 c.c. of water, add 25 c.c. of nitric acid, raise to the boiling point and aspirate air through the hot liquid until the liberated bromine is removed and the liquid is colourless. Cool and titrate against N/10 AgNO<sub>3</sub> using ferric ammonium sulphate as indicator. Not more than 3 c.c. should be required.

# 1 c.c. $N/10 \text{ AgNO}_3 \equiv 0.003546 \text{ gram Cl}$

### Organic Compounds

Shake 1 c.c. with 30 c.c. of water and a slight excess of ammonia, and allow to stand in a cool place for 2 hours. No oily drops should separate, and on evaporation to dryness a white residue should be left.

### Sulphate

The above residue dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 3 hours.

#### Arsenic

Limit 1 part per million.

Evaporate 10 grams, to which has been added 0·1 gram of anhydrous sodium carbonate and 0·2 c.c. of water, to dryness on a water-bath. Dissolve the residue in 50 c.c. of hot water, add 10 c.c. of hydrochloric acid and a few drops of stannous chloride solution and test as described on page 189.

# CADMIUM IODIDE A.R.

 $CdI_9 = 366 \cdot 27$ 

# Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )				10.0	per cent.
Iodate $(I_2O_5)$				0.002	per cent.
Alkalis and Alk	caline	Earths		0.2	per cent.

Pearly white flakes or a crystalline powder, readily soluble in water, forming a clear colourless solution.

# Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 1 hour.

#### Iodate

1 gram dissolved in 20 c.c. of water should not show any blue colour on addition of 1 c.c. of starch solution and 0·1 c.c. of N/1  $\rm H_2SO_4.$ 

# Alkalis and Alkaline Earths

Dissolve 1 gram in 50 c.c. of water, add 2 c.c. of ammonia, precipitate with hydrogen sulphide, filter and evaporate the filtrate to dryness, add 1 drop of sulphuric acid and ignite gently. The residue should not weigh more than 2 milligrams.

#### Assay

Dissolve 0.8 gram in water and titrate against N/10 AgNO<sub>3</sub> by Volhard's method.

1 c.c.  $N/10 \text{ AgNO}_3 \equiv 0.01831 \text{ gram CdI}_2$ 

Not less than 99 per cent, should be indicated.

# CADMIUM SULPHATE A.R.

 $CdSO_4 = 208 \cdot 47 + x H_2O$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.0005	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.002	per cent.
Iron (Fe)			0.0005	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )			0.00002	per cent.

Colourless crystals or crystalline powder, readily soluble in water.

# Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### 7---

1 gram dissolved in 10 c.c. of water and acidified with hydrochloric acid should not show more than a faint blue colour on addition of 1 c.c. of potassium ferrocyanide solution.

# Arsenic

Limit 0.2 part per million.

Dissolve 10 grams in 17 c.c. of water and 11 c.c. of hydrochloric acid, add 3 drops of stannous chloride solution and distil 20 c.c. To the distillate add 3 drops of stannous chloride solution and 40 c.c. of water, and test as described on page 189.

# CALCIUM CARBONATE A.R.

 $CaCO_3 = 100 \cdot 08$ 

# Maximum Limits of Impurities

Chloride (Cl) .			0.0002	per	cent.
Sulphate (SO <sub>3</sub> ) .					cent.
Phosphate (P <sub>2</sub> O <sub>5</sub> )			0.002	per	cent.
Heavy Metals and Ir	on		0.003	per	cent.
Magnesium (Mg).	,•		0.002	per	cent.
Alkalis			0.1	per	cent.
Acid-insoluble matter	: .		nil	_	

A white crystalline powder, almost insoluble in water; completely soluble with effervescence in dilute hydrochloric acid, forming a clear colourless solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and 2.5 c.c. of nitrie acid should not show any opalescence on addition of silver nitrate solution,

#### Sulphate

1 gram dissolved in 50 c.c. of water and 3 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 12 hours.

#### Phosphate

Dissolve 2 grams in 15 c.c. of dilute hydrochloric acid and 20 c.c. of water; add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B, and allow to stand for 5 minutes. No blue colour should be produced.

#### Heavy Metals and Iron

5 grams dissolved in a slight excess of dilute hydrochloric acid, boiled to remove carbon dioxide, and rendered alkaline with ammonia should not show more than a slight darkening on addition of 1 drop of sodium sulphide solution.

### Magnesium

Treat the solution from the last test with a slight excess of ammonium oxalate solution and stand on a hot plate for 3 hours. Filter, add sodium phosphate solution to the filtrate and allow to stand for 12 hours. No precipitate or turbidity should be produced.

(Continued overleaf)

#### Alkalis

Dissolve 5 grams in dilute nitric acid and precipitate with ammonia and ammonium carbonate. Filter and wash the precipitate. Evaporate the filtrate and washings to dryness and ignite gently; dissolve the residue in 1 c.c. of dilute nitric acid and 10 c.c. of water, add ammonia and ammonium oxalate, filter and evaporate the filtrate to dryness; add 2 drops of sulphuric acid, ignite gently, and weigh. Not more than 5 milligrams of residue should be left.

#### Assas

Dissolve 2 grams in 50 c.c. of N/1 HCl and 50 c.c. of water, and titrate the excess of acid against N/1 NaOH using bromophenol blue as indicator.

1 c.c. N/1 HCl = 0.05004 gram CaCO.

Not less than 99 per cent. should be indicated.

# CALCIUM CHLORIDE A.R.

 $CaCl_2 \cdot 6H_2O = 219 \cdot 09$ 

Maximum Limits of Impurities

Alcohol-insoluble matter		nil	
Sulphate (SO <sub>3</sub> )		0.005	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )		0.002	per cent.
Phosphate $(P_2O_5)$ .		0.002	per cent.
Heavy Metals and Iron		0.001	per cent.
Barium and Strontium.		10.0	per cent.
Alkalis		0.1	per cent.
Arsenic (As <sub>a</sub> O <sub>a</sub> )		0.0001	ner cent.

Colourless, deliquescent crystals, very soluble in water, forming a clear, colourless and neutral solution; soluble in alcohol.

### Sulphate

2 grams dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity or precipitate on addition of barium chloride solution and allowing to stand for 1 hour.

### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Phosphate

Dissolve 2 grams in 35 c.c. of water; add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B, and allow to stand for 5 minutes. No blue colour should be produced.

# Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Barium and Strontium

1 gram dissolved in 10 c.c. of water should not show any turbidity or precipitate on adding 10 c.c. of calcium sulphate solution and allowing to stand for 3 hours.

#### Albalia

Dissolve 5 grams in water and precipitate with ammonia and ammonium carbonate. Filter and wash the precipitate. Evaporate the filtrate and washings to dryness and ignite gently; dissolve the residue in 1 c.c. of dilute nitric acid and 10 c.c. of water, add ammonia and ammonium oxalate, filter and evaporate the filtrate to dryness; add 2 drops of sulphuric acid, ignite gently, and weigh. Not more than 5 milligrams of residue should be left.

#### Arsenic

Limit 1 part per million.

Test as described on page 189, using 5 grams with 10 c.c. of stannated hydrochloric acid.

# CARBON DISULPHIDE A.R.

 $CS_2 = 76 \cdot 12$ 

Maximum Limits of Impurities

Non-volatile residue . . . 0.003 per cent.

A clear, almost colourless, highly refractive, inflammable liquid with a characteristic odour. Insoluble in water, miscible with absolute alcohol and ether.

### Specific Gravity

About 1.27.

#### **Boiling Point**

46° to 47°.

### Residue

50 c.c. evaporated on a water-bath should not leave more than 2 milligrams of residue.

# CARBON TETRACHLORIDE A.R.

 $CCl_4 = 153 \cdot 83$ 

# Maximum Limits of Impurities

A clear, colourless liquid with a characteristic odour; almost insoluble in water; miscible with absolute alcohol and ether.

# Specific Gravity

About 1.60.

#### Boiling Range

76° to 77°.

#### Residue

20~c.c. evaporated on a water-bath should not leave more than 0.5~milligram of residue.

#### Free Chlorine

10 c.c. shaken with 5 c.c. of cadmium iodide and starch solution should not show a blue colour.

#### Fran Acid

Shake 10 c.c. with 10 c.c. of water for 1 minute; the aqueous layer should be neutral to litmus paper, and should not show any opalescence on addition of silver nitrate solution.

### Oxidisable Matter

Shake 10 c.c. with 10 c.c. of sulphuric acid and 10 c.c. of N/10  $\rm K_2Cr_2O_7$  for 10 minutes; dilute with 50 c.c. of water, cool, add 1 c.c. of potassium iodide solution and titrate the liberated iodine against N/10  $\rm Na_2S_2O_3$ . Not less than 9·5 c.c. should be required.

#### Carbon Disulphide

Boil 10 c.c. with 1 c.c. of absolute alcohol and 3 c.c. of potassium plumbite solution for 15 minutes under a reflux condenser, and allow to stand for 5 minutes. The aqueous layer should remain colourless.

# CHLORACETIC ACID

# $CH_2CICOOH = 94 \cdot 48$

# Maximum Limits of Impurities

Ash				0.05	per	cent.
Chloride (Cl)				0.003	per	cent.
Sulphate (SO <sub>3</sub> )				0.01	per	cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )				0.002	per	cent.
Heavy Metals ar	nd I	ron		0.001	per	cent.

Colourless crystals, readily soluble in water forming a clear solution.

#### Ach

2 grams should not leave more than 1 milligram of residue on ignition.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 20 c.c. of water should not show more than a faint turbidity on addition of barium chloride solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.e. of sulphuric acid.

#### Heavy Metals and Iron

1 gram dissolved in 30 e.c. of water should not show more than a faint darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Assay

Titrate about 3 grams dissolved in water against N/1 NaOH using phenolphthalein as indicator.

1 e.e., N/1 NaOH  $\equiv\!0\!\cdot\!09448$  gram CH<sub>2</sub>Cl . COOH

Not less than 99 per cent. should be indicated.

# CHLOROFORM A.R.

# CHCl<sub>2</sub> = 119.38

# Maximum Limits of Impurities

Non-volatile matt	er .		0.001 per cent.		
Free Acid .			no reaction		
Free Chlorine			no reaction		
Phosgene .			no reaction		
			no reaction		
Aldehyde .			no reaction		
Foreign organic	matter		no reaction		

A clear, colourless liquid with a characteristic odour; slightly soluble in water; miscible with alcohol and ether. Contains about 2 per cent, of alcohol as a preservative.

# Specific Gravity

# About 1.485.

# Boiling Point

#### About 61°.

# Residue

50 c.c. evaporated to dryness should not leave more than 1 milligram of residue.

#### Free Acid, Free Chlorine and Chloride

Shake 10 c.c. with 20 c.c. of  $CO_2$  free water and allow to separate. To 5 c.c. of the aqueous layer add  $0\cdot1$  c.c. of neutral litmus solution: the colour produced should not differ from that of 5 c.c. of CO, free water to which 0.1 c.c. of neutral litmus solution has been added. The addition of silver nitrate solution to another 5 c.c. of the aqueous layer should not produce any opalescence. The addition of 1 c.c. of cadmium iodide and starch solution to another 5 c.c. of the aqueous layer should produce no blue colour.

#### Hydrochloric Acid and Phosgene Decomposition Products

Mix 15 e.e. with 0.02 gram of vanillin and 0.02 gram of resorcinol and allow to stand in the dark for 1 hour, the solution should remain perfectly clear and colourless, and on shaking with 5 c.c. of 1 per cent. aqueous ammonia and allowing to separate, the aqueous layer should show no immediate pink colour.

### Ammonia and Aldehyde

Shake 10 c.c. with 20 c.c. of water, and separate. The aqueous portion should not give any appreciable colour on addition of either (1) Nessler's reagent or (2) Schiff's reagent.

### Foreign Organic Mutter

Shake 20 c.c. with 10 c.c. of sulphuric acid for 5 minutes and set aside in the dark for 30 minutes. No colour should be produced in either layer. Separate the two layers; dilute 2 c.c. of the acid layer with 5 c.c. of water, no unpleasant odour should be developed and on the further addition of 10 c.c. of water and 1 c.c. of N/10 AgNO $_3$  no opalescence should be produced. Shake 15 c.c. of the separated chloroform layer with 30 c.c. of water for 3 minutes, separate the aqueous layer and add to it 1 c.c. of N/10 AgNO $_3$ . No opalescence should be produced.

Shake 25 c.c. with 15 c.c. of sulphuric acid and 4 drops of formaldehyde solution and allow to stand for 1 hour in darkness. The acid should not be more than faintly coloured.

# CHROMIC ANHYDRIDE A.R.

(Chromic Acid)

 $\text{CrO}_3 = 100 \cdot 01$ 

Maximum Limits of Impurities

Sulphate Iron, Ali				0.02 per cent
salts Alkalis	:		:	no reaction 1.0 per cent.

Reddish-brown crystals or crystalline powder or almost black crystalline masses. Very soluble in water, forming a clear, orange-coloured solution.

### Sulphate

1 gram dissolved in 50 c.c. of water should not give any turbidity or precipitate on adding 1 c.c. of hydrochloric acid and 1 c.c. of barium chloride solution and allowing to stand for 3 hours.

### Iron, Aluminium and Chromium Salts

1 gram dissolved in 50 c.c. of water should form, on addition of excess of ammonia, a clear, pale yellow solution free from turbidity or precipitate.

#### Alkalis

Ignite 1 gram in a porcelain crucible, extract the residue with water and filter. The filtrate, evaporated to dryness, should not leave more than 10 milligrams of residue.

(Continued overleaf)

### Assay

Dissolve 0·1 gram in water, add potassium iodide solution and hydrochloric acid and titrate the liberated iodine against N/10  $Na_0S_2O_3$ .

1 e.e.  $N/10 Na_2S_2O_3 \equiv 0.003333 gram CrO_3$ 

Not less than 98 per cent, should be indicated.

# CHROMIUM POTASSIUM SULPHATE

(Chrome Alum)

 $CrK(SO_4)_2.12H_2O = 499.4$ 

#### Maximum Limits of Impurities

Chloride (Cl)			0.001	per	cent.
Ammonia (NH <sub>3</sub> )			0.01	per	cent.
Iron (Fe) .			O.I	per	cent.
Aluminium (Al)			0.1	per	cent.

Violet crystals, soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

### Ammonia

 $1\ \mathrm{gram}$  in powder heated with 10 c.c. of sodium hydroxide solution  $\cdot$  should not evolve any ammonia.

### Aluminium and Iron

Boil 1 gram with 10 c.c. of water, 5 c.c. of sodium hydroxide solution, and 10 c.c. of hydrogen peroxide (20 volumes), cool, and add 10 c.c. of ammonium chloride solution. Not more than a slight precipitate should be produced.

# CITRIC ACID

# $COOH.CH_2.C(OH)(COOH).CH_2.COOH.H_2O = 210.08$

# Maximum Limits of Impurities

Ash				0.01	per cent.
Sulphate (SO <sub>3</sub> )				0.002	per cent.
Oxalic Acid				no reac	tion
Tartaric Acid				no reac	tion
Heavy Metals an	d Iro	on .		0.0005	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )				0.0000	per cent.

Colourless crystals, readily soluble in water and in alcohol.

#### Asl

 $10~{
m grams}$  should not leave more than  $1~{
m milligram}$  of residue on ignition.

#### Sulphate

5 grams dissolved in 50 c.c. of water should not show any turbidity or precipitate on adding 1 c.c. of hydrochloric acid and 1 c.c. of barium chloride solution and allowing to stand for 12 hours.

#### Oxalic Acid

5 grams dissolved in 50 c.c. of water should not show any precipitate on adding 10 c.c. of ammonia and 2 c.c. of calcium chloride solution and allowing to stand for 1 hour.

#### Tartaric Acid

1 gram of the powdered acid heated with 10 c.c. of sulphuric acid on a water-bath for 30 minutes should not show any dark brown coloration.

#### Heavy Metals and Iron

10 grams dissolved in 30 c.c. of water and rendered alkaline with ammonia should not show more than a slight darkening on addition of 1 drop of sodium sulphide solution.

#### Arsenic

Limit 0·1 part per million.

Test as described on page 189, using 10 grams and 10 c.c. of stannated hydrochloric acid.

#### Assay

Dissolve 3 grams in water and titrate against  $N/1\ NaOH,$  using thymol blue as indicator.

1 c.c. N/1 NaOH  $\equiv 0.07003$  gram  $H_3C_6H_5O_7$ .  $H_9O$ 

Not less than 99.5 per cent. should be indicated.

# COBALT CHLORIDE A.R.

(Nickel and Iron free)

 $\mathrm{CoCl_2} \cdot 6\mathrm{H_2O} = 237 \cdot 95$ 

# Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )			o·oi per cent
Nickel (Ni) .			0.002 per cent
Iron (Fe)			0.003 per cent

Pink crystals, readily soluble in water forming a clear pink solution.

# Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Nickel

Dissolve 0·1 gram in 25 c.c. of water, add potassium cyanide solution until the precipitate formed is just redissolved; heat and shake the mixture for five minutes, then dilute to 50 c.c. with water at 85° and add 1 c.c. of 1 per cent. alcoholic dimethyl-glyoxime followed by dilute silver nitrate solution until a faint permanent precipitate is produced; allow to stand overnight. No red or pink colour should be produced.

### Iron

Dissolve 5 grams in water, add ammonium chloride and ammonia and filter through a small Buchner funnel. No brown residue should be left on the filter paper.

# COBALT NITRATE

(Nickel and Iron free)

 $\mathrm{Co(NO_3)_2.6H_2O} = 291 \!\cdot\! 05$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.003 per cent.
Sulphate (SO <sub>3</sub> )			0.01 per cent.
Nickel (Ni) .			0.002 per cent.
Iron (Fe) .			0.003 per cent.

Deliquescent red crystals, readily soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of 1 c.c. of silver nitrate solution,

### Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Nickel

Dissolve 0.1 gram in 25 c.c. of water, add potassium cyanide solution until the precipitate formed is just redissolved; heat and shake the mixture for five minutes, then dilute to 50 c.c. with water at 85° and add 1 c.c. of 1 per cent. alcoholic dimethyl-glyoxime followed by dilute silver nitrate solution until a faint permanent precipitate is produced and allow to stand overnight. No red or pink coloration should be produced.

#### Iro

Dissolve 5 grams in water, add ammonium chloride and ammonia and filter through a small Buchner funnel. No brown residue should be left on the filter paper.

# COPPER A.R.

Cu = 63.57

### Maximum Limits of Impurities

Acid-insolu	ıble :	matter		none
Tin .				no reaction
Lead .				no reaction
Silver .				no reaction
Iron (Fe)				0.005 per cent.
Arsenic (A	$s_2 O$	3)		0.00002 per cent.

Reddish metal needles or turnings.

#### Solubility

10 grams should dissolve completely in 35 c.c. of nitric acid diluted with 30 c.c. of water, forming a clear green solution.

#### Tin

Evaporate the above solution to dryness on a water-bath and dissolve the residue in 40 c.c. of water and 2 c.c. of nitric acid. The solution should be free from any insoluble residue.

#### I and

To 20 e.e. of the solution from the preceding test, add 1 e.e. of sulphuric acid and dilute with an equal volume of alcohol; no turbidity nor precipitate should be produced.

#### Silver

To the remainder of the solution from the tin test, add 1 c.c. of hydrochloric acid; no opalescence should be produced.

#### **7**....

To the solution from the above test add an excess of ammonia, filter through paper in a Buchner funnel of 5.5 cm. diameter, and wash the filter with dilute ammonia. Not more than a mere trace of brown residue should be left on the filter paper.

#### Arseni

Limit 2 parts per million.

Mix 5 grams with 4 grams of potassium chlorate and 15 c.c. of water, add 20 c.c. of hydrochloric acid in small portions at a time until all the copper is dissolved and boil gently to remove the bulk of the chlorine. Add 10 c.c. of water, 10 c.c. of hydrochloric acid, and a few drops of stannous chloride solution and distil 40 c.c.; to the distillate add 20 c.c. of water and a few drops of stannous chloride solution and test as described on page 189.

# COPPER ACETATE A.R.

 $(CH_3COO)_2Cu$ .  $H_2O = 199 \cdot 63$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.001	per cent.
Sulphate (SO <sub>3</sub> )			10.0	per cent.
Iron (Fe) .			0.01	per cent.

Dark green transparent crystals, slowly soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Iron

Boil 1 gram with 5 e.e. of water, 1 e.e. of hydrochloric acid, and 0·2 c.e. of nitric acid; dilute with 20 c.e. of water, make strongly alkaline with excess of ammonia, filter through a Buchner funnel of 5·5 cm. diameter and wash the filter paper with dilute ammonia Not more than a faint trace of a brown residue should be visible.

# Assay

Dissolve about 0·8 gram in 50 c.c. of water, acidify with acetic acid, add 3 grams of potassium iodide and titrate the liberated iodine with N/10  $\rm Na_2S_2O_3$ , using starch solution as indicator.

1 c.c. N/10  $I \equiv 0.01996 \text{ gram } (CH_3COO)_9Cu.H_9O$ 

Not less than 99 per cent. should be indicated,

# COPPER AMMONIUM CHLORIDÉ

 $CuCl_{2} \cdot 2NH_{4}Cl \cdot 2H_{9}O = 277 \cdot 51$ 

### Maximum Limits of Impurities

Free Acid .			absent
Sulphate (SO <sub>3</sub> )			0.005 per cent.
Iron (Fe) .			o·oɪ per cent.
Barium (Ba)			0.01 per cent.

Pale blue crystals, soluble in water, forming a clear solution which should be neutral to methyl orange.

#### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

#### Iros

1 gram dissolved in water, an excess of ammonia added, filtered on a Buchner funnel of 5.5 cm. diameter, and washed with dilute ammonia, should not leave more than a faint trace of a brown precipitate.

#### Barium

1 gram dissolved in 20 c.c. of water should not show any turbidity on adding 0.5 c.c. of dilute sulphuric acid and allowing to stand for one hour.

# COPPER CHLORIDE A.R. (Cupric)

 $CuCl_{2} \cdot 2II_{9}O = 170 \cdot 51$ 

### Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )				0.01	per cent.
Iron (Fe) .				0.028	per cent.
Barium (Ba)				0.01	per cent.
Alkalis and othe	r Me	tals		0.067	per cent.
Oxidisable matte	r (C	) .		0.003	per cent.
Arsenic (AsoCo)	``	٠	_	_	per cent.

Green or bluish-green crystals, very soluble in water forming a clear green or blue solution, according to dilution. Soluble in alcohol forming a clear solution.

#### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 5 minutes.

#### Iroz

Boil 5 grams with 10 c.c. of water and 1 c.c. of nitric acid; cool, add excess of ammonia, filter and wash with dilute ammonia. The presence of iron will be indicated by a brown precipitate. If present, dissolve in a little hydrochloric acid, reprecipitate with ammonia, filter off and ignite.

Not more than 2 milligrams of residue should be left.

#### Barium

1 gram dissolved in 20 c.c. of water should not show any turbidity on adding 0.5 c.c. of dilute sulphuric acid and allowing to stand for 1 hour.

#### Alkalis and other Metals

Dissolve 3 grams in water, acidify with hydrochloric acid, precipitate with hydrogen sulphide and filter. The filtrate, evaporated to dryness and ignited, should not leave more than 2 milligrams of residue.

#### Organic Matter

Digest 5 grams with 10 grams of silver sulphate and 50 c.c. of water on a water-bath until the reaction is complete, and filter. The filtrate, acidified with sulphuric acid, should not decolorise more than  $0.5\,\mathrm{c.c.}$  of N/10 KMnO<sub>4</sub>.

#### Arsenic

Limit 5 parts per million.

Dissolve 2 grams in 11 c.c. of hydrochloric acid and 7 c.c. of water, add 2 drops of stannous chloride solution and distil off 15 c.c. To the distillate add 45 c.c. of water and test as described on page 189.

#### Assav

Dissolve about 0.8 gram in 50 c.c. of water, acidify with acetic acid and add potassium iodide solution. Titrate the liberated iodine against  $N/10~Na_2S_2O_3$  using starch solution as indicator.

1 c.c. N/10 
$$Na_2S_2O_3 \equiv 0.01705$$
 gram  $CuCl_2 \cdot 2H_2O$ 

# COPPER CHLORIDE A.R. (Cuprous)

 $Cu_2Cl_2 = 198 \cdot 054$ 

# Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )		0.05 per cent
Iron (Fe)		0.014 per cent
Cupric chloride (CuCl <sub>2</sub> )		1.0 per cent
Arsenic (As <sub>0</sub> O <sub>0</sub> )		0.001 per cent

 $\Lambda$  greyish-white crystalline powder, soluble in concentrated hydrochloric acid and in ammonia.

1 gram boiled with 2 c.c. of nitric acid and 6 c.c. of water until dissolved, then diluted with 20 c.c. of water, should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Iras

Boil 5 grams with nitric acid, dilute with water and add excess of ammonia; filter and wash with dilute ammonia. The presence of iron will be indicated by a brown precipitate. If present, dissolve in a little hydrochloric acid and reprecipitate with ammonia, filter off and ignite. Not more than 1 milligram of residue should be left.

#### Cupric Chloride

Suspend 2 grams in 20 c.c. of water, add 5 c.c. of acetic acid and 2 grams of potassium iodide, and titrate the liberated iodine with N/10  $Na_2S_2O_3$  using starch solution as indicator.

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>  $\equiv$  0.01345 gram CuCl<sub>2</sub>

Not more than 1 per cent, should be indicated.

#### Arsenic

Limit 10 parts per million.

Dissolve 1 gram in 20 c.c. of 20 per cent. hydrochloric acid, add 2 drops of stannous chloride solution and distil 15 c.c. To the distillate add 45 c.c. of water and a few drops of stannous chloride solution and test as described on page 189.

# COPPER NITRATE A.R.

 $Cu(NO_3)_2.3H_2O = 241.63$ 

# Maximum Limits of Impurities

Chloride (Cl)				0.001	per cent.
Sulphate (SO <sub>3</sub> )	٠			0.01	per cent.
Iron (Fe) .				0.07	per cent.
Alkalis and other	r Mo	etals		0.2	per cent.

Blue crystals, readily soluble in water and in alcohol forming clear blue solutions.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of nitric acid should not show any turbidity on addition of 1 c.c. of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water should not show any turbidity or precipitate on adding 1 c.c. of barium chloride solution and allowing to stand for 1 hour.

#### Iron

Dissolve 5 grams in 15 c.c. of water, add excess of ammonia, filter and wash with dilute ammonia. The presence of iron will be indicated by a brown precipitate. If present, dissolve in a little hydrochloric acid, reprecipitate with ammonia, filter off and ignite. Not more than 5 milligrams of residue should be left.

#### Alkalis and other Metals

Dissolve 5 grams in 10 c.c. of water, add 2 c.c. of sulphuric acid and evaporate on a sand-bath until white fumes are evolved. Gool, dilute with water, remove the copper by precipitation with hydrogen sulphide and filter. The filtrate evaporated to dryness and ignited should not leave more than 10 milligrams of residue.

# Assay

Ignite about 1 gram and weigh the resulting CuO.

$$\frac{Cu(NO_3)_2.3H_2O}{CuO} \equiv 3.037$$

# COPPER OXIDE A.R. (Cupric)

 $CuO = 79 \cdot 57$ 

# Maximum Limits of Impurities

Chloride (Cl)				0.003	per cent.	
Sulphate (SO <sub>3</sub> )				0.03	per cent.	
Nitrate .				nil	•	
Carbonate .				nil		
Organic matter			,	nil		
Water-soluble:				0.05	per cent.	
Iron (Fe) .				0.1	per cent.	
Alkalis and oth	er Met	als		0.5	per cent.	

A black powder, insoluble in water, soluble in hydrochloric acid forming a clear green solution which remains clear on diluting with water and allowing to stand for some hours.

#### Chloride

1 gram dissolved in 3 c.c. of warm nitric acid and diluted with 30 c.c. of water should not show more than a faint opalescence on addition of silver nitrate solution.

# Sulphate

1 gram dissolved in 3 c.c. of warm hydrochloric acid and diluted with 30 c.c. of water should not show any turbidity on addition of 1 c.c. of barium chloride solution.

# Nitrate, Carbonate and Organic Matter

Ignite 5 grams in a combustion furnace in a stream of moist  $\mathrm{CO}_2$  free air; pass the issuing vapours over a piece of moist litmus paper and then through lime water. No change in either should take place.

# Water-soluble Matter

Boil 2 grams with 20 c.c. of water and filter; the filtrate should be neutral to litmus and on evaporation and ignition should not leave more than 1 milligram of residue.

#### Iron

Dissolve 2 grams in 10 c.c. of hydrochloric acid, warm with 1 c.c. of nitric acid, dilute with water and add an excess of ammonia, filter and wash with dilute ammonia. The presence of iron will be indicated by a brown precipitate. If present, dissolve in a little hydrochloric acid, reprecipitate with ammonia, filter off and ignite. Not more than 3 milligrams of residue should be left.

#### Alkalis and other Metals

Dissolve 2 grams in 10 c.c. of hydrochloric acid, dilute with water, precipitate the copper with hydrogen sulphide and filter; the filtrate evaporated to dryness and ignited should not leave more than 10 milligrams of residue.

# COPPER SULPHATE A.R.

 $CuSO_4.5H_2O = 249.71$ 

### Maximum Limits of Impurities

Chloride (Cl)				0.001	per	cent.
Iron (Fe) .				0.014	per	cent.
Alkalis and oth	ier Me	tals		0.1	per	cent.

Blue crystals, soluble in water forming a clear blue solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Iron

Boil 5 grams with 15 c.c. of water and 1 c.c. of nitric acid, cool, add excess of ammonia, filter and wash with dilute ammonia. The presence of iron will be indicated by a brown precipitate. If present, dissolve in a little hydrochloric acid, reprecipitate with ammonia, filter off and ignite. Not more than 1 milligram of residue should be left.

#### Alkalis and other Metals

Dissolve 5 grams in water, acidify with hydrochloric acid, rentove the copper by precipitation with hydrogen sulphide and filter. The filtrate evaporated to dryness and ignited should not leave more than 5 milligrams of residue.

#### Assay

Dissolve 1 gram in water, acidify with acetic acid and add potassium iodide solution. Titrate the liberated iodine against N/10  $Na_9S_9O_3$  using starch as indicator.

1 c.c. N/10  $Na_2S_2O_3 \equiv 0.02497$  gram  $CuSO_4$ .  $5H_2O$ 

Not less than 99 per cent. should be indicated.

# DEXTROSE A.R.

 $C_6H_{12}O_6 = 180 \cdot 09$ 

### Maximum Limits of Impurities

Alcor	ioi-ins	oluble	matte	er.		nıl	
Ash						0.04	per cent.
	ure						per cent.
Acidi	ty, 100	g. ≡	not	more	than	0.3 c.c.	N/I.
Chlor	ride (C	1)				0.001	per cent.
Heav	y Meta	ıls and	Iron			0.0005	per cent.
	ite					no reacti	on
Arsen	ic (As	$_2O_3$				0.00005	per cent.

A white powder, very soluble in water forming a clear colourless solution.

#### Ash

10 grams should not leave more than 4 milligrams of residue on ignition.

#### Moisture

5 grams should not lose more than 10 milligrams on drying at 100°.

10 grams dissolved in 100 c.c. of hot water should not require more than 0.3 c.c. of N/10 NaOH to produce a pink colour with phonolphthalein.

5 grams dissolved in 100 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Heavy Metals and Iron

2 grams dissolved in 50 c.c. of water and rendered alkaline with ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Arsenic and Sulphite

Limit of arsenic 0.5 part per million.

Test as described on page 189 using 10 grams and 10 c.c. of stannated hydrochloric acid. The lead paper should not show more darkening than one used in a test from which the dextrose is omitted.

#### Dextrin

I gram should dissolve in 30 e.c. of boiling ethyl alcohol (90 per cent.) and form a clear solution.

Specific Rotation [a]  $^{20}_{\ \ D}^{\circ}$  not less than + 52°.

# DIMETHYL-AMINO-AZOBENZENE A.R.

(Dimethyl yellow)

 $C_6H_5N : NC_6H_4 . N(CH_3)_2 = 225 \cdot 14$ 

Maximum Limits of Impurities

0.2 per cent. Alcohol-insoluble matter

Golden brown crystals, almost insoluble in water; soluble in alcohol to a clear orange or yellow solution.

#### Melting Point

116° to 118°.

# pH Range

2.8 to 4.0.

1 gram should not leave more than 2 milligrams of residue on ignition.

# Sensitivity

One drop of a 0·2 per cent, solution in alcohol added to 20 c.c. of water should colour the liquid yellow and should be changed to bright red by the addition of 1 drop of N/10 HCl.

# p-DIMETHYLAMINOBENZALDEHYDE A.R.

 $(CH_3)_2N \cdot C_6H_4 \cdot CHO = 149 \cdot 09$ 

Maximum Limits of Impurities

Sulphated ash 0.05 per cent. Organic impurities passes test

A pale yellow crystalline powder, readily soluble in alcohol and in dilute hydrochloric acid forming clear solutions.

(Continued overleaf)

Melting Point 73° to 75°.

#### Ach

2 grams moistened with sulphuric acid should not leave more than 1 milligram of residue on ignition.

#### Organic Impurities

25 milligrams dissolved in 10 c.c. of sulphuric acid should produce an almost colourless solution.

# DIMETHYLANILINE A.R.

 $C_6H_5N(CH_3)_2 = 121 \cdot 09$ 

Maximum Limits of Impurities

Aniline and Methylaniline . . 0.2 per cent.

Completely soluble in dilute hydrochloric acid

An almost colourless oily liquid when freshly distilled, but which rapidly darkens to a reddish brown colour; slightly soluble in water.

Specific Gravity
About 0.960.

Freezing Point
About 2.5°.

Boiling Range 192° to 194°.

#### Solubility

 $5\,\mathrm{c.c.}$  should dissolve to a clear solution in  $30\,\mathrm{c.c.}$  of dilute hydrochloric acid.

#### Aniline and Methylaniline

To 10 grams, add 20 c.c. of a 10 per cent. solution of acetic anhydride in benzene, allow to stand in a stoppered flask for 30 minutes, add 50 c.c. of water, shake well and titrate against N/1 NaOH, using phenolphthalein as indicator. In the same manner titrate a blank on 20 c.c. of the acetic anhydride solution. The difference between the titrations should not exceed  $0\cdot 2$  c.c.

# DIMETHYLGLYOXIME & A.R.

# $\text{CH}_3.\text{C(NOH)}.\text{C(NOH)}.\text{CH}_3 = 116.08$

# Maximum Limits of Impurities

Ash .					0.05 per cent.
Alcohol-in	nsolub	le ma	tter		nil
Purity					passes quantitative limit
					toct

A white crystalline powder, soluble in alcohol forming a clear solution; almost insoluble in water.

#### Melting Point

About 240° with decomposition.

#### Ash

1 gram should not leave more than 0.5 milligram of residue on ignition.

#### Limit of Purity

Dissolve 0·24 gram of crystallised nickel chloride (NiCl $_2$ ·6H $_2$ O) in 100 c.c. of water, heat to boiling, and add 0·25 gram of the dimethylglyoxime dissolved in 25 c.c. of 90 per cent. alcohol. Add ammonia drop by drop until alkaline, cool and filter. To the filtrate add 5 c.c. of 1 per cent. solution of dimethylglyoxime in 90 per cent. alcohol, heat to boiling and cool. No red precipitate should be produced.

# DIPHENYLAMINE A.R.

 $(C_6H_5)_2NH = 169 \cdot 09$ 

# Maximum Limits of Impurities

Sulphated ash . . . . 0.02 per cent. Nitrate . . . absent

White crystals, slightly soluble in water; readily soluble in alcohol and in ether.

# Melting Point

54° to 55°.

### Sulphated Ash

2 grams moistened with sulphuric acid should not leave any appreciable residue on ignition.

#### Nitrate

2 milligrams dissolved in a cooled mixture of 6 c.c. of sulphuric acid, 2 c.c. of water and 1 drop of hydrochloric acid should form a perfectly colourless solution.

#### Sensitivity

To the solution from the above test add  $0.1\,\mathrm{c.c.}$  of a solution containing 0.002 milligram of potassium nitrate and allow to stand for 5 minutes. A distinct blue colour should be produced.

# ESCHKA'S MIXTURE A.R.

Maximum Limits of Impurities

Sulphate (SO<sub>3</sub>) . . . o · o · per cent.

A mixture of 1 part of sodium carbonate A.R. and 2 parts of magnesium oxide A.R.

#### Sulphate

2 grams dissolved in 9 c.c. of hydrochloric acid and 50 c.c. of water should not show any turbidity or precipitate on adding 1 c.c. of barium chloride solution and allowing to stand for 1 hour.

# ETHER A.R.

 $(C_2H_5)_2O = 74 \cdot 08$ 

#### Maximum Limits of Impurities

Non-volatile ma	tter				o·ooi per cent.
Free Acid .					no reaction
Water					passes test
Aldehyde, Aceto	ne ai	ıd Vir	ıyl co	m-	•
pounds .			٠.		o·oooi per cent.
Peroxide (calcula	ited 2	s H.C	1.0	_	0.000005 per cent.

A clear, colourless, mobile, inflammable liquid with a characteristic odour; soluble in water (1 in 10), miscible with alcohol and other organic solvents.

Specific Gravity
About 0.720.

Boiling Point

34° to 35°.

#### Residue

20 c.c. should not leave a visible residue on evaporation.

#### Free Acid

Heat 20 c.c. with 5 c.c. of water on a water-bath and continue the heat for 5 minutes after the ether has evaporated, then add 5 drops of neutral methyl red solution. The colour should not differ from that of 5 c.c. of water to which 5 drops of the methyl red solution have been added.

#### Water

Should form a clear liquid with an equal volume of carbon disulphide.

# Aldehyde, Acetone and Vinyl Compounds

Place 5 c.c. of Nessler reagent in a 30 c.c. stoppered bottle and fill the bottle completely with the ether. Insert the stopper, shake well, and allow to stand for 5 minutes. No colour nor turbidity should be produced.

#### Peroxide

Place 10 c.c. of ferrous thiocyanate reagent in a 30 c.c. stoppered bottle previously filled with carbon dioxide; completely fill the bottle with the ether, insert the stopper, shake and allow to stand for 5 minutes in the dark. No pink colour should be produced.

# ETHYL ALCOHOL A.R.

(Absolute Alcohol)

 $C_0H_cOH = 46.05$ 

# Maximum Limits of Impurities

Residue				nil
Fusel Oil				passes test
Aldehyde			•	passes test
Tannin				no reaction

A clear, colourless liquid, miscible with water without opalescence, the solution being neutral to litmus.

Specific Gravity

0.794 to 0.797.

**Boiling Range** 

77.5° to 78.5°.

#### Residue

10 c.c. should not leave any residue on evaporation.

#### Fusel Oil

5 c.c. poured on to filter paper and allowed to evaporate spontaneously should not afford a foreign odour at any stage of the evaporation.

# Aldehyde, Tannin, etc.

10 c.c. should not show an immediate darkening on mixing with 5 c.c. of sodium hydroxide solution.

10 c.c. should not show an immediate darkening on mixing with 5 c.c. of ammonia.

Mix, at 15°, 1 c.c. with 1 c.c. of Schiff's reagent and keep at that temperature for 10 minutes, and add 4 c.c. of water. The colour viewed in a  $2\cdot 5$  cm. cell should not be deeper than  $3\cdot 5$  red  $+1\cdot 0$  blue (Lovibond).

# ETHYL ALCOHOL A.R.

(90 per cent.)

#### Maximum Limits of Impurities

Residue				nil
Fusel Oil				passes test
Aldehyde				passes test
, Tannin		•		no reaction

# Specific Gravity

0.832 to 0.835.

This should conform to the qualitative tests in the preceding monograph.

# FERRIC AMMONIUM SULPHATE

 $FeNH_4(SO_4)_2 . 12II_2O = 482 \cdot 19$ 

Maximum Limits of Impurities

Chloride (Cl) . . . . o · oot per cent. Ferrous salt . . . no reaction

Pale violet or almost colourless crystals, readily soluble in water forming a clear pale brown solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

# Ferrous Salt

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any blue or green colour on addition of 1 drop of potassium ferricyanide solution.

#### Assay

Dissolve about 2 grams in 60 c.c. of water, add 20 c.c. of hydrochloric acid and 3 grams of potassium iodide and titrate the liberated iodine against N/10 Na<sub>9</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>2</sub> = 0.04822 gram FeNH<sub>4</sub>(SO<sub>4</sub>)<sub>2</sub>.  $12H_2O$ 

Not less than 99 per cent. nor more than 101 per cent. should be indicated.

# FERRIC CHLORIDE (ANHYDROUS) A.R.

 $FeCl_3 = 162 \cdot 21$ 

# Maximum Limits of Impurities

Insoluble matter .			nil
Sulphate (SO <sub>3</sub> ) .			o.oo6 per cent.
Nitrate $(N_2O_5)$ .			0.002 per cent.
TO COLL 1 /CD			0.001 per cent.
Ferrous salt (Fe)			0.002 per cent.
Alkalis and other Meta	ls		0.04 per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .			0.005 per cent.
Copper (Cu) .			0.005 per cent.

A greenish-black crystalline powder, very hygroscopic, becoming orange coloured on hydration. Readily soluble in its own weight of water forming a clear solution.

# Sulphate

2 grams dissolved in 50 e.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding barium chloride solution and standing for 6 hours.

Dissolve 1 gram in 10 c.c. of water, add excess of ammonia and filter, neutralise the filtrate with dilute sulphuric acid, add 0.5 c.c. of indigo solution and an equal volume of sulphuric acid. The blue colour should not be discharged.

# Free Chlorine

Boil 1 gram with 10 c.c. of water and expose starch iodide paper to the vapours. No blue colour should be produced.

### Ferrous Salt

0.2 gram dissolved in 10 c.c. of water should not show a blue colour on addition of 10 c.c. of potassium ferricyanide solution.

# Alkalis and other Metals

Dissolve 5 grams in 100 c.c. of water, add excess of ammonia and filter. The filtrate should be colourless. One half of the filtrate evaporated to dryness and ignited should not leave more than 1 milligram of residue. To the other half add ammonium sulphide solution. No precipitate should be produced.

#### Arsenic

Limit 50 parts per million.

Dissolve I gram in 11 c.c. of hydrochloric acid and 7 c.c. of water. add stannous chloride solution until the iron is completely reduced to the ferrous state as shown by the colour of the solution. Distil 15 c.c. and to the distillate (or a portion of it) add 50 c.c. of water, a few drops of stannous chloride solution, more hydrochloric acid if necessary, and test as described on page 189,

# FERROUS AMMONIUM SULPHATE A.R.

 $FeSO_4.(NH_4)_9SO_4.6H_9O = 392 \cdot 13$ 

Contains not less than 99.9 per cent. nor more than 100.1 per cent. of FeSO<sub>4</sub>.(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O.
Pale greenish-blue crystals or a crystalline powder.

Soluble in water forming a clear solution.

#### Assay

Dissolve 1.9607 grams in water, acidify with dilute sulphuric acid and titrate against N/10 K2Cr2O7 using potassium ferricyanide as external indicator.

1 c.c N/10 
$$K_2Cr_2O_7 \equiv 0.03921$$
 gram  $FcSO_4.(NH_4)_2SO_4.6H_2O$ 

Not less than 99.9 per cent, nor more than 100.1 per cent, should be indicated.

# FERROUS SULPHATE

 $FeSO_4.7H_2O = 278 \cdot 01$ 

Maximum Limits of Impurities

Insoluble in Water			nil	
Heavy Metals .			0.001	per cent.
Free Acid (H <sub>2</sub> SO <sub>4</sub> )			0.05	per cent.
Alkalis and other Met	als		0.04	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .			0.0001	per cent.

Green or bluish-green crystals, readily soluble in freshly boiled and cooled water forming a clear solution.

(Continued overleaf)

#### Alkalis and other Metals

Dissolve 5 grams in 50 c.c. of water, add 5 c.c. of nitric acid, boil, precipitate with ammonia and filter. The filtrate should be colourless, and one half evaporated to dryness and ignited should not leave more than 1 milligram of residue. To the other half add ammonium sulphide solution; no precipitate should be produced.

# Free Acid

5 grams dissolved in 50 c.e. of water should not require more than 0.5 c.c. of  $N/10\,$  NaOH to produce a yellow colour with methyl orange.

### Heavy Metals

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of dilute sulphuric acid, should not show any darkening in colour or precipitate on saturating with hydrogen sulphide.

#### Arsenic

Limit 1 part per million.

Dissolve 5 grams in 11 c.c. of hydrochloric acid and 7 c.c. of water, add a few drops of stannous chloride solution and distil 13 c.c. To the distillate add 50 c.c. of water and a few drops of stannous chloride solution and test as described on page 189.

#### Assay

Dissolve about 1 gram in water, acidify with dilute sulphuric acid and titrate against N/10  $\rm K_2Cr_2O_7$  using potassium ferricyanide as external indicator.

1 c.c. N/10  $K_2Cr_2O_7 \equiv 0.02780$  gram FeSO<sub>4</sub>.7H<sub>2</sub>O

Not less than 99 per cent. should be indicated.

# FORMALDEHYDE SOLUTION A.R.

HCHO = 30.02

#### Maximum Limits of Impurities

Non-volatile matter			0.0025 per cent.
Free Acid (HCOOH)			o·14 per cent.
Chloride (Cl) .			o.ooo3 per cent.
Heavy Metals .			o.oooi per cent.
Organic impurities	_		nassés test

A clear, colourless liquid with a strong pungent odour; miscible with water and with alcohol forming clear solutions.

#### Residue

20 c.c. evaporated to dryness and ignited gently should not leave more than 0.5 milligram of residue.

10 c.c. require for neutralisation not more than 0.3 c.c. N/1 NaOH using phenolphthalein as indicator.

10 c.c. diluted with 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

5 c.c. diluted with 45 c.c. of water should not show any coloration on addition of ammonia and 1 drop of sodium sulphide solution.

#### Organic Impurities

10 c.c. mixed with 10 c.c. of N/1 NaOH and allowed to stand for 1 hour should remain colourless.

Mix 3 c.c. with 50 c.c. of N/1 NaOH and add 50 c.c. of 3 per cent. hydrogen peroxide; warm on a water-bath for 30 minutes with occasional shaking; titrate the excess of alkali against N/1  $\rm H_2SO_4$ using phenolphthalcin as indicator.

At the same time carry out a blank determination omitting the

formaldehyde solution.

Each c.c. difference between the titrations corresponds to 1 per cent. weight/volume of CH2O.

Not less than 37 per cent. weight/volume should be indicated.

# FORMIC ACID A.R.

(98/100 per cent.)

# $HCOOH = 46 \cdot 02$

Maximum Limits of Impurities

Non-volatile matter			o·or per cent.
Chloride (Cl) .			o·oo1 per cent.
Sulphate (SO <sub>3</sub> ) .			0.005 per cent.
Heavy Metals and Iro	n.		o · ooı per cent.

A clear, colourless liquid with a pungent odour; miscible with water forming a clear, colourless solution which should not show any turbidity on standing for half an hour.

(Continued overleaf)

# Specific Gravity

About 1.225.

#### Residue

10 c.c. evaporated to dryness should not leave more than 1 milligram of residue.

#### Chloride

1 c.c. diluted with 10 c.c. of water should not show more than a slight opalescence on addition of silver nitrate solution and nitric add

### Sulphate

2 c.c. diluted with 10 c.c. of water should not show any turbidity on addition of barium chloride solution.

#### Heavy Metals and Iron

2 c.c. diluted with 40 c.c. of water should not show more than a faint darkening on addition of ammonia and 1 drop of sodium sulphide solution.

# Assay

Titrate 2 grams dissolved in water against  $\rm N/1$  NaOH using phenolphthalein as indicator.

# 1 c.c. N/1 NaOH $\equiv 0.04602$ gram HCOOH

Not less than 98 per cent. should be indicated.

# FORMIC ACID A.R.

Maximum Limits of Impurities

Non-volatile matter			o·oi per cent.
Chloride (Cl) .			o·ooi per cent.
Sulphate $(SO_3)$ .		•	0.005 per cent.
Heavy Metals and Iron	١.		o · ooı per cent.

### Specific Gravity

About 1·20.

This should conform to the limit tests in the preceding monograph.

# FUSION MIXTURE A.R.

# Maximum Limits of Impurities

Chloride (Cl)			0.002	per	cent.
Sulphate (SO <sub>3</sub> )			0.03	per	cent.
Nitrate $(N_2O_5)$			0.002	per	cent.
Heavy Metals and	Iron		0.0005	per	cent.

An equimolecular mixture of anhydrous potassium and sodium carbonates; soluble in water forming a clear colourless solution,

#### Chloride

2 grams dissolved in 50 c.c. of water and acidified with 3 c.c. of nitric acid should not show more than a faint opalescence on addition of 1 c.c. of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 9 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and standing for 6 hours.

#### Nitrate

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

# Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water should not show any darkening on addition of 1 drop of sodium sulphide solution.

# GLYCEROL A.R.

# $CH_2OH$ . CHOH . $CH_2OH = 92.06$

#### Maximum Limits of Impurities

Non-volatile matter	•		٠	0.008	per	cent.	
				0.0005	per	cent.	
Sulphate (SO <sub>3</sub> ) .				0.001	per	cent.	•
Volatile Fatty Esters	(as	Glyce	eryl		-		
Butyrate) .				0.025	per	cent.	
Heavy Metals and Iron				0.0001	per	cent.	
Carbonisable impuritie	s			nil	-		
Reducing substances				nil			
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .				0.0002	per	cent.	

A clear, colourless, syrupy liquid with a sweet taste. Very hygroscopic, miscible with water and alcohol.

# Specific Gravity

1.260 to 1.264.

M --- 1--1.

# Residue and Sugars

20 c.c. ignited in a platinum dish should not leave more than 2 milligrams of residue, and in dissipating the last traces of glycerol not more than a slight charring should occur and there should be no odour of burnt sugar.

#### Chlorida

5 c.c. diluted with 20 c.c. of water should not show any opalescence on addition of silver nitrate solution,

#### Sulphate

5 c.c. diluted with 20 c.c. of water should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

### Fatty Acids

5 c.c. warmed with 5 c.c. of dilute sulphuric acid and vigorously shaken should not develop more than a faint unpleasant odour.

#### Volatile Fatty Esters

Boil 20 grams with 90 c.c. of  $\mathrm{CO_2}$  free water and 2 c.c. of sodium hydroxide solution (50 per cent.) for 2 minutes, add 50 c.c. of dilute sulphuric acid (2.5 per cent. by volume) and distil 110 c.c. Titrate the distillate against N/10 NaOH, using phenolphthalein as indicator. Not more than 0.5 c.c. N/10 NaOH should be required.

10 c.c. diluted with 40 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

10 c.c. diluted with 40 c.c. of water should not assume more than a faint transient pink or purple colour on addition of 1 drop of ammonia and 1 drop of tannic acid solution (10 per cent.).

#### Organic Impurities

10 c.c. mixed with 10 c.c. of sulphuric acid, keeping cold the while, should not become more than straw coloured.

Reducing Substances 5 c.c. diluted with 5 c.c. of ammonia should not darken in colour on adding a few drops of silver nitrate solution and keeping in darkness for 5 minutes.

Limit 2 parts per million.

Test as described on page 189, using 5 grams and 10 c.c. of stannated hydrochloric acid.

# GUANIDINE CARBONATE A.R.

 $(NH_2)_2$ :C:NH]<sub>2</sub>.H<sub>2</sub>CO<sub>3</sub> = 180·14

## Maximum Limits of Impurities

Ash			0.3	per cent.	
Chloride (Cl)			0.000	7 per cent.	
Sulphate (SO <sub>3</sub> )			0.002	per cent.	

A white crystalline powder, soluble in water forming a clear colourless solution.

1 gram moistened with sulphuric acid and ignited should not leave more than 2 milligrams of residue.

1 gram dissolved in 10 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

(Continued overleaf)

# HYDRIODIC ACID

Sp. Gr. 1.94

 $HI = 127 \cdot 94$ 

A clear liquid varying in colour from pale yellow to dark brown, the colour being due to free iodine.

#### Assay

Dilute 3 to 4 grams with water, titrate any free iodine against  $N/10 \text{ Na}_2S_2O_3$  and then add phenolphthalein and titrate the acidity against N/1 NaOH.

1 c.c. N/1 NaOH = 0.12794 gram HI

About 66 per cent. should be indicated.

# HYDROBROMIC ACID

(30 per cent.)

 $HBr = 80 \cdot 92$ 

### Maximum Limits of Impurities

Non-volatile matter .		0.03	per cent.
Hydrochloric Acid (HCl)		O·I	per cent.
Sulphuric Acid (SO <sub>3</sub> ) .		0.008	per cent.
Heavy Metals and Iron .		0.0005	per cent.
Arsenic (As <sub>0</sub> O <sub>0</sub> )		0.0005	per cent.

A clear, colourless or faintly yellow liquid, miscible with water and alcohol.

Specific Gravity

About 1.265.

#### Residue

10 c.c. evaporated to dryness and ignited gently should not leave more than 3 milligrams of residue.

# Hydrochloric Acid

Mix 10 c.c. with 65 c.c. of water and 25 c.c. of nitric acid, boil gently and bubble air through until the liberated bromine is removed and the liquid is colourless. Cool and titrate against  $N/10~{\rm AgNO_{2}}$ .

1 c.c. AgNO<sub>3</sub> = 0.003646 gram HCl

Not more than 0.1 per cent. should be indicated.

### Sulphuric Acid

5 c.c. diluted with 50 c.c. of water should not show more than a faint turbidity on addition of barium chloride solution.

### Heavy Metals and Iron

5 c.c. diluted with 20 c.c. of water and rendered alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

# Arsenia

Limit 5 parts per million.

Test as described on page 189, using 2 grams and 10 c.c. of stannated hydrochloric acid.

#### Assav

Titrate 5 grams diluted with water against N/1 NaOH using methyl red as indicator.

1 c.c. N/1 NaOH  $\equiv 0.08092$  gram HBr

Not less than 30 per cent. should be indicated.

# HYDROCHLORIC ACID

 $HCl = 36 \cdot 46$ 

# Maximum Limits of Impurities

Non-volatile matter		0.0017	per cent.
Sulphuric Acid (SO <sub>3</sub> )		0.0004	per cent.
Heavy Metals .		0.0002	per cent.
Iron (Fe)		0.0002	per cent.
Free Chlorine (Cl)		0.0002	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .		0.00004	per cent.

A clear, colourless fuming liquid.

# Specific Gravity

About 1.18.

#### Residue

50 e.c. evaporated to dryness on a water-bath and ignited gently should not leave more than 1 milligram of residue.

(Continued overleaf)

#### Sulphuric Acid

Evaporate 50 c.c. on a water-bath until reduced to about 5 c.c., add 100 c.c. of water and 5 c.c. of barium chloride solution and allow to stand for 12 hours. No precipitate or turbidity should be produced.

#### Heavy Metals

5 c.c. diluted with 20 c.c. of water should not show more than a faint darkening on addition of 20 c.c. of hydrogen sulphide water.

#### Iron

2 c.c. diluted with 20 c.c. of water should not show more than a faint blue colour on addition of 1 c.c. of potassium ferrocyanide solution and allowing to stand for 30 minutes.

#### Free Chlorine

2 c.c. diluted with 20 c.c. of water should not show any blue colour on addition of cadmium iodide and starch solutions.

#### Arsenic

Limit 0.04 part per million.

Evaporate 50 c.c. with a few drops of bromine solution on a water-bath until reduced to 15 c.c., add 50 c.c. of hot water and a few drops of stannous chloride solution, and test as described on page 189.

#### Assay

Titrate 3-4 grams diluted with water against N/1 NaOH using methyl red as indicator.

1 c.c. N/1 NaOH = 0.03646 gram HCl

About 36 per cent. should be indicated.

# HYDROFLUORIC ACID

 $HF = 20 \cdot 0$ 

### Maximum Limits of Impurities

		0.02	per cent.
Silica		passes	test
Sulphuric Acid (SO <sub>3</sub> )		0.01	per cent.
Heavy Metals and Iron		0.001	per cent.

A colourless fuming liquid, strongly corrosive.

#### Residue

10 c.c. evaporated in a platinum dish and ignited gently should not leave more than 2 milligrams of residue.

#### C:1:--

2 c.c. diluted with 10 c.c. of water should not give any turbidity or precipitate on addition of 5 c.c. of 20 per cent. potassium chloride solution.

# Sulphuric Acid

2 c.c. evaporated in a platinum dish on a water-bath and the residue diluted with 10 c.c. of water should not give an immediate precipitate on addition of 1 c.c. of barium chloride solution.

#### Heavy Metals and Iron

5 c.c. diluted with 20 c.c. of water and made slightly alkaline with ammonia should be almost colourless, and should not show more than a slight darkening on addition of one drop of sodium sulphide solution.

#### Assay

Weigh a flask and 50 c.c. of N/1 NaOH, add about one gram of the acid, reweigh, and titrate the excess of alkali against N/1  $\rm H_2SO_4$  using methyl red as indicator.

1 c.c. N/1 NaOH  $\equiv 0.02001$  gram HF

Not less than 40 per cent. should be indicated.

# HYDRÔGEN PEROXIDE ◆ A.R.

(20 volumes)

 $H_2O_2 = 34 \cdot 02$ 

# Maximum Limits of Impurities

Non-volatile matter			o·oi per cen	t.
Acidity, 100 c.c. = not	more	than	2 c.c. N/10	
Chloride (Cl) .			o·oooi per cen	t.
Sulphate (SO <sub>3</sub> ) .			0.001 per cen	t.
Phosphate (P2O5)			0.001 per cen	t.
Barium (Ba)			0.003 per cen	ıt.

A clear, colourless liquid.

#### Residue

 $10\ \mathrm{c.c.}$  evaporated to dryness on a water-bath should not leave more than 1 milligram of residue.

(Continued overleaf)

#### Acidity

 $10\,c.c.$  diluted with  $20\,$  c.c. of water should not require more than  $0.2\,c.c.$  of  $N/10\,$  NaOH to neutralise the free acid, methyl orange being used as indicator.

#### Chloride

 $10\,c.c.$  diluted with  $10\,c.c.$  of water and acidified with  $0.5\,c.c.$  of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

10 c.c. diluted with 10 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 1 hour.

#### Phosphate

Evaporate 5 c.c. to dryness and dissolve the residue (if any) in 5 c.c. of dilute sulphuric acid and 30 c.c. of water, then add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B. No blue or green colour should appear.

#### Barium

 $10~\rm c.c.$  diluted with  $10~\rm c.c.$  of water should not show any turbidity on adding dilute sulphuric acid and allowing to stand for 6 hours.

#### Assav

Titrate 2 c.c. diluted with 20 c.c. of water and acidified with dilute sulphuric acid against N/10 KMnO<sub>4</sub>.

1 c.c. N/10 KMnO<sub>4</sub>  $\equiv$  0 · 283 volume  $\equiv$  0 · 0017 gram H<sub>2</sub>O<sub>2</sub>

Not less than 6 per cent. (20 volumes) should be indicated.

# HYDROGEN PEROXIDE ◆○→ A.R.

(10 volumes)

 $H_2O_2 = 34 \cdot 02$ 

# Maximum Limits of Impurities

Non-volatile matter			o·oi per cent.
Acidity, 100 c.c. ≡ not	more	e than	2 c.c. N/10
Chloride (Cl) .			o·ooo1 per cent.
Sulphate $(SO_3)$ .			o·ooi per cent.
Phosphate (P2O5)			0.001 per cent.
Barium (Ba) .			0.003 per cent.

This should conform to the tests for purity in the preceding monograph.

# HYDROXYLAMINE HYDROCHLORIDE A.R.

 $NH_2OH \cdot HCl = 69 \cdot 50$ 

Maximum Limits of Impurities

Colourless crystals, very soluble in water forming a clear, colourless solution; completely soluble in alcohol.

#### Ash

1 gram should not leave more than  $0\!\cdot\! 5$  milligram of residue on ignition.

# Sulphate

 $2\ grams\ dissolved\ in\ 20\ c.c.$  of water should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

#### Ammonia

1 gram boiled with 5 c.c. of water and 5 c.c. of sodium hydroxide solution should not evolve any ammoniaeal odour.

## Assay

Dissolve  $0\cdot 1$  gram in 10 c.c. of water, add 60 c.c. of 10 per centsolution of ferric ammonium sulphate and 15 c.c. of dilute sulphuric acid, and boil gently for 5 minutes; cool, and titrate against N/10 KMnO<sub>4</sub>.

1 e.e.  $N/10~KMnO_4 \equiv 0.003475~gram~NH_2OH$ . HCl

Not less than 98 per cent, should be indicated.

# 8-HYDROXYQUINOLINE A.R.

 $C_9H_6N \cdot OH = 145 \cdot 06$ 

# Maximum Limits of Impurities

Ash			0.05 per cei	nt.
Sulphate (SO <sub>3</sub> )			0.02 per cer	nt.
Chloride (Cl)			0.001 per cer	nt.

Almost white crystals or crystalline powder. Soluble in alcohol and in dilute ammonia forming clear almost colourless solutions. Soluble in dilute hydrochloric acid forming a clear yellow solution.

#### Melting Point

74° to 76°.

#### Ash

 ${f 2}$  grams should not leave more than 1 milligram of residue on ignition.

#### Sulphate

1 gram dissolved in 1 c.c. of hydrochloric acid and 30 c.c. of water should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and 1 c.c. of nitric acid should not show any opalescence on addition of 1 c.c. of silver nitrate solution.

#### Assay

Dissolve about 0·15 gram in 50 c.c. of water and 20 c.c. of hydrochloric acid, add 50 c.c. of N/10 bromide-bromate solution, allow to stand for 5 minutes; dilute to 200 c.c. with water, add 10 c.c. of potassium iodide solution and titrate with N/10  $\rm Na_2 S_2 O_3$ .

1 e.c. N/10 bromide-bromate  $\equiv\!0\!\cdot\!003627$  gram  $C_9H_6N$  , OH

Not less than 99 per cent. should be indicated.

# IODIC ACID

# $\mathrm{HIO_3} = 175 \! \cdot \! 94$

# Maximum Limits of Impurities

Sulphated ash			0.05 per cent.
Insoluble matter			nil
Sulphate (SO <sub>2</sub> )			0.05 per cent.

A white or almost white powder, readily soluble in an equal weight of water.

#### Sulphated Ash

2 grams moistened with sulphuric acid and ignited should not leave more than 1 milligram of residue.

#### Sulphate

Boil gently 1 gram with 1 gram of granulated zinc, 45 c.c. of water and 5 c.c. of hydrochloric acid. When the reaction has ceased, cool, nearly neutralise with ammonia, add 1 c.c. of barium chloride solution and allow to stand for 1 hour. No turbidity should be produced.

#### Assav

Dissolve 0·1 gram in 20 c.c. of water, add 5 c.c. of potassium iodide solution and 2 c.c. of hydrochloric acid and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 e.c. N/10 Na $_2{\rm S}_2{\rm O}_3\!\equiv\!0\!\cdot\!002932$  gram  ${\rm HIO}_3$ 

Not less than 99 per cent, should be indicated.

# IODINE A.R.

 $I = 126 \cdot 932$ 

#### Maximum Limits of Impurities

Ash . . . . . o·oɪ per cent. Insoluble matter . . . nil

Bluish-black crystalline scales with a metallic lustre, slightly soluble in water, soluble in organic solvents and in solutions of alkali iodides.

#### Act

 $10\ \mathrm{grams}$  should not leave more than  $1\ \mathrm{milligram}$  of residue on ignition.

#### Solubility

5 grams dissolved in potassium iodide solution, and excess of sodium thiosulphate solution (20 per cent.) added, should form a clear, colourless solution.

#### Assan

Dissolve 0.5 gram in potassium iodide solution, and titrate against N/10 Na\_2S\_2O\_3.

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>  $\equiv$  0 · 01269 gram I

Not less than 99.9 per cent, should be indicated.

# LACTIC ACID

 $\mathrm{CH_3}$  .  $\mathrm{CHOH}$  .  $\mathrm{COOH} = 90 \cdot 05$ 

# Maximum Limits of Impurities

Ash				0.02	per cent.
Chloride (Cl)				0.002	per cent.
Sulphate (SO <sub>3</sub> )				0.005	per cent.
Heavy Metals an	d Ir	on.		0.001	per cent.
Arsenic (As <sub>2</sub> O <sub>2</sub> )				0.0001	per cent.

 ${\bf A}$  clear, syrupy liquid, almost colourless and odourless. Miscible with water, alcohol, and ether. Insoluble in chloroform.

#### Aci

 $10~{\rm grams}$  should not leave more than 5 milligrams of residue on ignition.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

I gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution and allowing to stand for 2 hours.

#### Heavy Metals and fron

1 gram dissolved in 40 c.c. of water and rendered alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Arsenic

Limit 1 part per million.

Test as described on page 189 using 10 grams with 10 c.c. of stannated hydrochloric acid.

#### Estimation

Dissolve about 4 grams in 100 c.c. of water, add 50 c.c. of N/1 NaOH, boil gently for 10 minutes and titrate the excess of alkali against N/1 H<sub>2</sub>SO<sub>4</sub> using phenolphthalein as indicator.

1 c.c. N/1 NaOH = 
$$0.09005$$
 gram  $C_3H_6O_3$ 

Not less than the equivalent of 87 per cent. of  $\mathrm{C_3H_6O_3}$  should be indicated.

# LACTOSE A.R.

 $C_{12}H_{22}O_{11}$ .  $H_2O = 360 \cdot 19$ 

# Maximum Limits of Impurities

Ash		0.05 per cent.
Moisture		0.4 per cent.
Acidity ( $C_3H_6O_3$ )		o·o45 per cent.
Sucrose and Dextrose		o·1 per cent.
Formaldehyde .		nil
Nitrogen (N) .		0.05 per cent.

A white crystalline powder, slightly soluble in cold water, more readily in hot water, forming a clear, colourless solution.

#### Asl

10 grams should not leave more than 5 milligrams of residue on ignition.

#### Moisture

5 grams should not lose more than 20 milligrams on drying at 100° for 1 hour.

#### Acidity

10 grams dissolved in 100 c.c. of hot water should not require more than 0.5 c.c. N/10 NaOH to produce  $\epsilon$  pink colour with phenolphthalein.

#### Sucrose and Dextrose

Shake 5 grams with 20 c.c. of 90 per cent. alcohol for 30 minutes and filter. 10 c.c. of the filtrate evaporated to dryness should not leave more than 5 milligrams of residue.

# Formaldehyde

1 gram dissolved in 10 c.c. of water should not show a violet colour on addition of 1 c.c. of Schiff's reagent.

#### Nitrogen

5 grams oxidised by Kjeldahl's method and distilled with sodium hydroxide should not produce more ammonia than will be neutralised by 1.8 c.c. of N/10  $H_2SO_4$ .

# LEAD ACETATE A.R.

 $(CH_3COO)_9Pb.3H_2O = 379.31$ 

# Maximum Limits of Impurities

Insoluble matter			nil
Chloride (Cl)			0.0005 per cent
Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.002 per cent
Copper (Cu)			o.ooi per cent
Iron (Fe) .			0.001 per cent
Alkalis .			0.05 per cent

White crystalline masses, readily soluble in water with at most a slight opalescence, which is removed by the addition of a few drops of acetic acid.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should produce a precipitate having a blue tint on addition of 10 c.c. of sulphuric acid.

#### Copper

Dissolve 1 gram in 15 c.c. of water, add 5 c.c. of ammonium acetate solution and 2 c.c. of acetic acid. Then add 3 drops of pyridine, 5 c.c of ammonium thiocyanate solution and 2 c.c. of chloroform; shake vigorously and allow to separate. The chloroformic layer should not be coloured green.

#### Iron

Dissolve 1 gram in 40 c.c. of water and add 5 c.c. of nitric acid and 1 c.c. of ammonium thiocyanate solution; not more than a faint pink colour should be produced.

#### Albali

Dissolve 10 grams in 200 c.c. of water, add 10 c.c. of hydrochloric acid and filter, remove the remainder of the lead by means of hydrogen sulphide, filter, evaporate the filtrate to dryness, add 1 drop of sulphuric acid, and ignite. Not more than 5 milligrams of residue should be left.

### LEAD ACETATE BASIC

Approximate formula (CH<sub>3</sub>COO)<sub>2</sub>Pb.PbO

### Maximum Limits of Impurities

Nitrate (N <sub>2</sub> O <sub>5</sub> )			o·oo1 per cent.
Copper (Cu)			0.002 per cent.
Iron (Fe) .			0.002 per cent.

A heavy white powder, slowly soluble in water forming a slightly hazy alkaline solution. Soluble in glycerol.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 1 c.c. of glacial acetic acid, and 1 c.c. of indigo solution added, should produce a precipitate having a blue tint on addition of 10 c.c. of sulphuric acid.

#### Copper

Dissolve 0.5 gram in 15 c.e. of hot water, add 5 c.c. of ammonium acetate solution and 2 c.c. of acetic acid and cool. Then add 3 drops of pyridine, 5 c.c. of ammonium thiocyanate solution and 2 c.c. of chloroform; shake vigorously and allow to separate. The chloroformic layer should not be coloured green.

#### Iron

Dissolve 0.5 gram in 40 c.c. of water and add 5 c.c. of nitric acid and 1 c.c. of ammonium thiocyanate solution; not more than a faint pink colour should be produced.

#### Assay for Total Lead

Dissolve 0.5 gram in 50 c.c. of water, acidify with acetic acid, heat nearly to boiling and precipitate with 1 gram of oxalic acid. Cool, filter, and wash the precipitate free from soluble oxalate, suspend it in 50 c.c. of water, acidify with sulphuric acid, warm to  $60^{\circ}$  and titrate with  $N/10\ KMnO_{4}$ .

1 c.e. N/10 KMnO<sub>4</sub> = 0.01371 gram (CH<sub>3</sub>COO)<sub>2</sub>Pb.PbO

Not less than 95 per cent. should be indicated.

#### Assay for Alkalinity

Dissolve 5 grams in 100 c.c. of water, add 50 c.c. of N/1  $\rm H_{2}SO_{4}$  and sufficient water to produce 200 c.c. Shake, allow to settle, decant 100 c.c. of the clear liquid and titrate the excess of acid with N/1 NaOH using phenolphthalein as indicator.

1 c.c. N/1  $H_9SO_4 \equiv 0.2742$  gram  $(CH_3COO)_9Pb.PbO$ 

Not less than 90.0 per cent, should be indicated.

### LEAD DIOXIDE

 $PbO_9 = 239 \cdot 22$ 

Maximum Limits of Impurities

Water-soluble matter . . . o · 1 per cent. Chloride (Cl) . . . . o · 002 per cent. Manganese . . . no reaction

A dark brown amorphous powder, insoluble in water.

### Chloride

1 gram boiled with 1 c.c. of nitric acid and 20 c.c. of water and filtered should not show more than a faint opalescence on addition of silver nitrate solution.

### Water-soluble Matte

Boil 2 grams with water and filter; the filtrate evaporated to dryness should not leave more than 2 milligrams of residue,

#### Manganese

Boil 2 grams with 5 c.c. of nitric acid and 1 c.c. of water, cool, add 15 c.c. of water and 5 c.c. of dilute sulphuric acid and allow the precipitate to settle. The supernatant liquid should not be coloured pink.

#### Assay

Shake 0.5 gram with 1 gram of potassium iodide, 25 grams of sodium chloride, 100 c.c. of water and 20 c.c. of hydrochloric acid in a stoppered flask for 5 minutes, and titrate the liberated iodine against  $N/10~Na_2S_2O_3$  using starch as indicator.

1 c.c.  $N/10 \text{ Na}_2S_2O_3 \equiv 0.01196 \text{ gram PbO}_2$ 

Not less than 95 per cent, should be indicated.

### LEAD NITRATE (60 H) A.R.

### $Pb(NO_3)_2 = 331 \cdot 23$

## Maximum Limits of Impurities

Insoluble matter				none
Moisture .				o·1 per cent.
Copper (Cu)				o.ooi per cent.
Iron (Fe) .				o.ooi per cent.
Alkalie and other	Meta	le		O'T per cent.

White crystals or crystalline powder, soluble in water forming a clear colourless solution.

#### Moisture

5 grams dried at 100° should not lose more than 5 milligrams.

#### Copper

Dissolve 1 gram in 15 c.c. of hot water, add 5 c.c. of ammonium acetate solution and 2 c.c. of acetic acid and cool. Then add 3 drops of pyridine, 5 c.c. of ammonium thiocyanate solution and 2 c.c. of chloroform; shake vigorously and allow to separate. The chloroformic layer should not be coloured green.

#### Iron

Dissolve 1 gram in 40 c.c. of water and add 5 c.c. of nitric acid and 1 c.c. of ammonium thiocyanate solution; not more than a faint pink colour should be produced.

### Alkalis and other Metals

Dissolve 5 grams in 100 c.c. of water, add 2 grams of ammonium acetate and 5 c.c. of acetic acid and precipitate the lead with hydrogen sulphide, filter and evaporate the filtrate to dryness and ignite. The residue should not weigh more than 5 milligrams.

### LEAD OXIDE A.R.

PbO == 223 · 22

### Maximum Limits of Impurities

Insoluble in Acetic Acid		0.1	per cent.
Chloride (Cl)		0.003	per cent.
Nitrate $(N_2O_5)$		0.003	per cent.
Copper (Cu)		0.001	per cent.
Iron (Fe)		0.001	per cent.
Silver (Ag)		0.0002	per cent.
Alkalis and Alkaline Earths		0.3	per cent.

A heavy orange coloured powder, insoluble in water; readily soluble in dilute acctic acid and in warm solutions of alkali hydroxides.

#### Insoluble Matter

Treat 20 grams with warm acetic acid diluted with an equal volume of water, filter and wash the filter paper with water until free from acid. The residue after drying should not weigh more than 0.02 gram.

#### Chloride

1 gram dissolved in 2 c.c. of nitric acid and 50 c.c. of water should not show more than a faint opalescenec on addition of 1 c.c. of silver nitrate solution.

#### Nitrate

1 gram dissolved in 5 c.c. of acetic acid, and 5 c.c. of water and 0.5 c.c. of indigo solution added, should produce a precipitate having a blue tint on addition of 10 c.c. of sulphuric acid.

#### Copper

Dissolve 1 gram in 5 c.c. of acetic acid and 15 c.c. of water, add 5 c.c. of ammonium acetate solution, 3 drops of pyridine, 5 c.c. of ammonium thiocyanate solution and 2 c.c. of chloroform; shake vigorously and allow to separate. The chloroformic layer should not be coloured green.

#### Iron

Dissolve 1 gram in 5 c.c. of nitric acid and 40 c.c. of water and add 1 c.c. of ammonium thiocyanate solution; not more than a faint pink colour should be produced.

#### Silver

Dissolve 20 grams in 30 c.c. of nitric acid and 100 c.c. of boiling water, add a mixture of 10 c.c. of sulphuric acid and 10 c.c. of water, mix thoroughly and evaporate until white fumes are evolved. Cool, dilute with 100 c.c. of 50 per cent. alcohol and filter; to the filtrate add 1 c.c. of dilute hydrochloric acid; no opalescence should be produced.

#### Alkalis and Alkaline Earths

Dissolve 10 grams in 20 c.c. of acetic acid and 50 c.c. of water, add 10 c.c. of hydrochloric acid and filter. Remove the lead from the filtrate by means of hydrogen sulphide and again filter. The filtrate evaporated to dryness and ignited with 1 drop of sulphuric acid should not leave more than 0.03 gram of residue.

### MAGNESIUM AMMONIUM CHLORIDE A.R.

 $MgCl_2 \cdot NH_4Cl \cdot 6H_2O = 256 \cdot 82$ 

### Maximum Limits of Impurities

		0.02	per cent.
		0.002	per cent.
Calcium (Ca) .		0.005	per cent.
Heavy Metals and Iron		0.001	per cent.
Barium (Ba) .		0.002	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .		0.0005	per cent.

White crystals, readily soluble in water forming a clear, colourless solution.

### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.4 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

#### Phosphate

 $5~\rm grams$  dissolved in 50 c.c. of water should not show any turbidity on addition of 1 c.c. of ammonia.

#### Calcium

1 gram dissolved in 20 c.c. of water should not show any turbidity on addition of 1 c.c. of ammonium oxalate solution.

#### Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water should not show any appreciable darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Barium

1 gram dissolved in 20 c.c. of water should not show any turbidity on adding dilute sulphuric acid and allowing to stand for 6 hours.

# on addi

Limit 5 parts per million.

Test as described on page 189 using 2 grams with 10 c.c. of stannated hydrochloric acid.

#### Assay

Titrate 0.4 gram dissolved in water against N/10 AgNO<sub>3</sub> by Volhard's method.

1 c.c. N/10 AgNO<sub>3</sub> 
$$\equiv$$
 0·008561 gram MgCl<sub>2</sub> . NH<sub>4</sub>Cl . 6H<sub>2</sub>O

Precipitate 1·0 gram by means of sodium phosphate and ammonia and weigh as  ${\rm Mg_2P_2O_7}$ .

$$\frac{2MgCl_2\cdot NH_4Cl\cdot 6H_2O}{Mg_2P_2O_7} \equiv 2\cdot 3065$$

Not less than 96 per cent. should be indicated.

### MAGNESIUM CHLORIDE

 $MgCl_2$ .  $6H_2O = 203 \cdot 33$ 

#### Maximum Limits of Impurities

Alcohol-insoluble matte	r .		nil		
Sulphate (SO <sub>3</sub> )			0.006	per	cent.
Phosphate (P2O5)			0.0005	per	cent.
Heavy Metals and Iron			0.001		
Calcium (Ca)			0.002		
Barium (Ba) .			0.01	per	cent.
Arsenic (As <sub>2</sub> O <sub>2</sub> ) .	,		0.0005	per	cent.

Colourless deliquescent crystals. Readily soluble in less than its own weight of water forming a clear, colourless solution, and soluble in 6 times its weight of 90 per cent. alcohol.

#### Sulphate

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 1 hour.

#### Phosphate

Dissolve 2 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B, and allow to stand for 5 minutes. No blue colour should be produced.

#### Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water should not darken in colour on addition of ammonium chloride solution, ammonia, and 1 drop of sodium sulphide solution.

#### Calcium

The above solution should not show any turbidity on the further addition of a few drops of ammonium oxalate solution.

#### Barium

1 gram dissolved in 20 e.e. of water should not show any turbidity on adding sulphuric acid and allowing to stand for 6 hours.

#### Arsenic

Limit 5 parts per million.

Test as described on page 189 using 2 grams with 10 c.c. of stannated hydrochloric acid.

#### Assay

Titrate 0.5 gram dissolved in water against  $N/10~\Lambda gNO_3$  by Volhard's method.

1 e.e.  $N/10 \text{ AgNO}_3 \equiv 0.01017 \text{ gram MgCl}_2 \cdot 6H_2O$ 

### MAGNESIUM OXIDE

 $MgO = 40 \cdot 32$ 

### Maximum Limits of Impurities

Loss on ignition				5.0	per cent.	
Chloride (Cl)				0.003	per cent.	
Sulphate (SO <sub>3</sub> )				0.01	per cent.	
Nitrate (N <sub>2</sub> O <sub>5</sub> )				0.003	per cent.	
Carbonate .				trace		
Heavy Metals and	Iron	١.		0.002	per cent.	
Arsenic (As <sub>0</sub> O <sub>0</sub> )				0.0001	per cent.	

A white powder, almost insoluble in water, readily soluble in dilute acids forming clear solutions.

#### Moisture, etc.

 $2\,$  grams heated to dull redness should not lose more than  $100\,$  milligrams in weight.

#### Chlorida

1 gram dissolved in 50 c.c. of dilute nitric acid should not show more than a slight opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 6 c.c. of hydrochloric acid and 40 c.c. of water should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 1 hour.

#### Nitrate

1 gram dissolved in 25 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 25 c.c. of sulphuric acid.

### Carbonate

I gram boiled with 50 c.c. of water for 5 minutes and cooled should not give more than a faint effervescence on addition of 10 c.c. of hydrochloric acid.

#### Heavy Metals and Iron

1 gram dissolved in 25 c.c. of acetic acid and made alkaline with ammonia should not show more than a slight darkening on addition of 5 drops of sodium sulphide solution.

#### Arsenic

Limit 1 part per million.

Dissolve 5 grams in 40 c.c. of brominated hydrochloric acid and 20 c.c. of water, add a few drops of stannous chloride solution and test as described on page 189.

#### MAGNESIUM SULPHATE A.R.

 ${
m MgSO_4}$  ,  $7{
m H_2O}=246\cdot49$ 

#### Maximum Limits of Impurities

Reaction .			neutral
Chloride (Cl)			0.0002 per cent.
Phosphate (P2O5)			o · ooo5 per cent
Heavy Metals			o·0005 per cent.
Iron (Fe) .			o·0005 per cent.
Zinc (Zn) .			o·0005 per cent.
Arsenic (As <sub>0</sub> O <sub>2</sub> )			o·oooi per cent.

Colourless crystals, readily soluble in water forming a clear, colourless solution, neutral to litmus.

#### Chloride

1 gram dissolved in 10 c.c. of water and acidified with 0.2 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Phosphate

Dissolve 2 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B, and allow to stand for 5 minutes. No blue colour should be produced.

#### Heavy Metals

2 grams dissolved in 20 c.c. of water, and 1 c.c. of glacial acetic acid added, followed by a slight excess of ammonia, should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Iron and Zinc

5 grams dissolved in 50 c.c. of water should not show any opalescence or more than a faint blue colour on adding 1 c.c. of hydrochloric acid and 1 c.c. of potassium ferrocyanide solution and allowing to stand for 1 hour.

#### Arsenic

Limit 1 part per million.

Dissolve 10 grams in 50 c.c. of water, add 10 c.c. of stannated hydrochloric acid and test as described on page 189.

### MANGANESE CHLORIDE

 $MnCl_2.4H_2O = 197.91$ 

#### Maximum Limits of Impurities

Sulp	hate (S	$O_3$					0.01	per	cent.
Iron	(Fe)						0.0008	per	cent.
Zinc	(Zn)						0.05	per	cent.
Barit	ım (Ba	)					0.02	per	cent.
Alka	lis						0.1	per	cent.
Oxid	lising ar	nd re	ducing	g subs	tances	s(O)	0.0004	per	cent.

Pink crystals, readily oxidising on exposure to air, becoming brown. Very soluble in water forming a clear, pink solution.

#### Sulphate

2 grams dissolved in 50 c.c. of water and acidified with I c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

#### Iron

1 gram dissolved in 20 c.c. of water, acidified with hydrochloric acid, boiled with 1 c.c. of chlorine water and cooled should not show any red colour on addition of potassium thiocyanate solution.

#### Zinc

1 gram dissolved in 20 c.c. of water and acidified with acetic acid should not give any precipitate on addition of hydrogen sulphide water.

#### Barium

1 gram dissolved in 20 c.c. of water should not show any turbidity on adding 1 c.c. of dilute sulphuric acid and allowing to stand for 2 hours.

#### Alkalis

Dissolve 2 grams in 50 c.c. of water and precipitate the manganese by means of ammonium carbonate. Filter and wash. The filtrate evaporated to dryness and ignited should not leave more than 2 milligrams of residue.

### Oxidising and Reducing Substances

5 grams dissolved in 50 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show any blue colour on addition of potassium iodide and starch solution, and the further addition of 1 drop of N/10~I should produce a blue colour.

### MANGANESE SULPHATE A.R.

 $MnSO_4.4H_2O=223\cdot05$ 

#### Maximum Limits of Impurities

Chloride (Cl)			•	0.002	per cent.
Iron (Fe) .				0.0008	per cent.
Zinc (Zn) .				0.05	per cent.
Calcium (Ca)				0.02	per cent.
Magnesium an	d Alka	lis .		0.1	per cent.
Oxygen absort	bed (O	) .		0.0008	per cent.

Pinkish transparent crystals or a white powder, readily soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### 7---

1 gram dissolved in 20 c.c. of water, acidified with 1 c.c. of hydrochloric acid, boiled with 1 c.c. of chlorine water and cooled should not show any red colour on addition of potassium thiocyanate solution.

#### Zinc

1 gram dissolved in 20 c.c. of water, acidified with 1 c.c. of acetic acid and 1 gram of sodium acetate added, should not give any precipitate on addition of 20 c.c. of hydrogen sulphide water.

#### Calcium

1 gram dissolved in 50 c.c. of water should not show any turbidity on addition of 1 c.c. of ammonium oxalate solution.

#### Magnesium and Alkalis

Dissolve 2 grams in 50 c.e. of water, precipitate the manganese with ammonium carbonate and filter. The filtrate evaporated to dryness and ignited should not leave more than 2 milligrams of residue.

#### Reducing Substances

10 grams dissolved in 100 c.c. of cold water and acidified with sulphuric acid should not require more than 0.1 c.c. N/10 KMnO<sub>4</sub> to produce a pink colour.

### MERCURIC CHLORIDE A.R.

 $\mathrm{HgCl_2} = 271 \cdot 52$ 

Maximum Limits of Impurities

Other Metals . . . 0 °02 per cent.

Arsenic . . . no reaction

Completely soluble in water, in alcohol, and in ether

Heavy colourless crystals, soluble in water, in alcohol and in ether forming clear, colourless solutions.

#### Other Metals

Dissolve 5 grams in 100 c.c. of water, acidify with hydrochloric acid, precipitate with hydrogen sulphide and filter. The filtrate evaporated to dryness and ignited gently should not leave more than 1 milligram of residue.

#### Arcenic

Digest the precipitate obtained in the above test with 5 c.c. of ammonia and 5 c.c. of water, filter and acidify the filtrate with hydrochloric acid. No yellow precipitate should be obtained.

#### Assay

Treat 5 grams in a tared beaker with a slight excess of hypophosphorous acid, heat on a water-bath until the mercury appears as a globule, decant the acid liquor, wash well with water, then with alcohol and finally with ether, dry for a few minutes in a warm cupboard and weigh.

$$\frac{HgCl_2}{Hg}\equiv\!1\cdot\!3536$$

Not less than 99.5 per cent. should be indicated.

### MERCURIC OXIDE A.R. (RED)

 $HgO = 216 \cdot 6$ 

#### Maximum Limits of Impurities

$\mathbf{A}$ sh			0.1	per cent.
Chloride (Cl)			0.005	per cent.
Sulphate (SO <sub>2</sub> )			0.01	per cent.

A red crystalline powder, insoluble in water, soluble in mineral acids forming mercuric salts.

#### Asl

 $1\ \mathrm{gram}$  should not leave more than  $1\ \mathrm{milligram}$  of residue on ignition.

#### Chloride

1 gram dissolved in 2 c.c. of nitric acid and 20 c.c. of water should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 2 c.c. of hydrochloric acid and 20 c.c. of water should not show any turbidity on addition of barium chloride solution.

#### Assav

Dissolve about 0.5 gram in 5 c.c. of nitric acid, dilute with 50 c.c. of water and titrate against N/10 NII<sub>4</sub>SCN using ferric alum as indicator.

1 c.e. N/10 NH<sub>4</sub>SCN  $\equiv$  0·01083 gram HgO

Not less than 99.6 per cent. should be indicated.

### MERCUROUS CHLORIDE A.R.

 $HgCl = 236 \cdot 07$ 

Maximum Limits of Impurities

Ash		0.05	per cent.
Mercuric salt (Hg)		0.001	per cent.
Sulphate (SO <sub>2</sub> ) .		0.02	per cent.

#### Ash

 $5~{\rm grams}$  should not leave more than  $1~{\rm milligram}$  of residue on ignition (in a fume closet).

#### Mercuric Salt

1 gram shaken with 50 c.c. of cold water and filtered should not show any colour on addition of 1 drop of sodium sulphide solution.

#### Sulphate

I gram shaken with 20 c.c. of water and filtered should not show any turbidity on addition of barium chloride solution.

### MERCUROUS NITRATE A.R.

 $HgNO_3.H_2O = 280.62$ 

Maximum Limits of Impurities

Acid-insoluble matte	r.		nil
<b>A</b> sh			0.05 per cent.
Sulphate (SO <sub>2</sub> )			O'I per cent.

Colourless or opaque white crystals, readily soluble in dilute nitric acid forming a clear solution.

#### Ash

2 grams gently ignited in a fume closet should not leave more than 1 milligram of residue.

#### Sulphate

2 grams dissolved in 20 e.c. of water and  $5 \, \mathrm{c.c.}$  of nitric acid should not show any turbidity on addition of 1 e.c. of barium nitrate solution.

#### Assay •

Treat about 1 gram with 3 c.c. of acctic acid, 1 gram of sodium acetate, 10 c.c. of water, 50 c.c. of N/10 I, and 1 gram of potassium iodide. Shake occasionally until solution is complete and titrate the excess of iodine against N/10  $\rm Na_2S_2O_3$ .

1 e.c. N/10  $I \equiv 0.02806$  gram of  $HgNO_3.H_2O$ 

Not less than 95 per cent. should be indicated.

### MERCURY A.R. (REDISTILLED)

 $\mathrm{Hg} = 200 \cdot 61$ 

### Maximum Limits of Impurities

Non-volatile matter . . . 0 · 002 per cent. Acid-insoluble matter . . . nil

Other Metals . . . no reaction

A liquid metal with a brilliant silvery lustre.

#### Non-volatile Matter

 $50~{\rm grams}$  evaporated in a fume closet should not leave more than 1 milligram of residue.

### Acid-insoluble Matter

10 grams should dissolve completely on warming with 30 c.c. of nitric acid and 30 c.c. of water.

#### Other Metals

Heat 5 grams in a boiling water-bath for one minute with 5 grams of sodium thiosulphate dissolved in 5 c.c. of water. The surface of the mercury should not be tarnished.

### METHYL ALCOHOL A.R.

 $CH_2OH = 32 \cdot 03$ 

Maximum Limits of Impurities

Non-volatile matter		o ooi per cent.
Carbonisable matter		none
Acetone		o·o4 per cent.
Organic impurities		passes tests

A clear colourless liquid, miscible with water.

#### Specific Gravity

About 0.796.

#### **Boiling Point**

65° to 66°.

#### Residue

20 c.c. evaporated on a water-bath should not leave any residue.

#### Organic Impurities

When mixed with an equal volume of sulphuric acid, keeping cool the while, not more than the faintest yellow colour should be produced.

When mixed with an equal volume of sodium hydroxide solution no colour should be produced.

#### Acetone

Add 5 c.c. of the alcohol to a mixture of 10 c.c. of N/1 NaOH and 30 c.c. of water, adjust to 15° and add 25 c.c. of N/10 I and allow to stand for 30 minutes at 15°; add 15 c.c. of N/1 H<sub>2</sub>SO<sub>4</sub> and titrate the excess of iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

From the volume of N/10 I absorbed, deduct 0.5 c.c. representing the average error of the method.

1 e.e.  $N/10 I \equiv 0.0009677 \text{ gram } (CH_3)_2CO$ 

Not more than 0.04 per cent. should be indicated.

### MOLYBDIC ACID A.R.

 $MoO_3 = 144 \cdot 0$  $H_2MoO_4 = 162 \cdot 01$ 

#### Maximum Limits of Impurities

Insoluble in Ammonia		nil	
Chloride (Cl)		0.005	per cent
Sulphate (SO <sub>3</sub> ) .		0.12	per cent
Phosphate (P2O5)		0.0005	per cent
Heavy Metals		0:0005	ner cent

A white or pale cream-coloured powder, readily and completely soluble in ammonia.

#### Chloride

1 gram dissolved in 10 c.c. of water and 3 c.c. of ammonia should not show any opalescence on acidifying with nitric acid and adding silver nitrate solution.

#### Sulphate

1 gram dissolved in 10 c.c. of water and 3 c.c. of ammonia should not give any precipitate on acidifying with nitric acid, adding barium nitrate solution and standing for 6 hours.

#### Phosphate

Dissolve 0.2 gram in 1 c.c. of ammonia solution and add 50c.c. of N/1 H<sub>2</sub>SO<sub>4</sub> and 5 c.c. of phosphate reagent B. No blue colour should be produced in 5 minutes.

#### Heavy Metals

2 grams dissolved in 20 c.c. of ammonia should not give any brown coloration on addition of 1 drop of sodium sulphide solution.

#### Assar

Dissolve about 0.2 grams in 5 c.c. of water and a few drops of ammonia solution and add 95 c.c. of N/1  $\rm H_2SO_4$ . Pass this solution very slowly through a long glass tube containing amalgamated zinc needle turnings, and from which the air has been displaced by N/1  $\rm H_2SO_4$ . Allow the end of the tube to dip into a mixture of 10 c.c. of 10 per cent. solution of ferric ammonium sulphate and 10 c.c. of dilute sulphuric acid. Wash the tube through with 50 c.c. of N/1  $\rm H_2SO_4$  and titrate the solution with N/10 KMnO<sub>4</sub>.

1 c.c.  $N/10 \text{ KMnO}_4 \equiv 0.0048 \text{ gram MoO}_3$ 

Not less than 85 per cent, of  $\mathrm{MoO_3}$  should be indicated.

### 

 $MoO_3 = 144 \cdot 0$ 

### Maximum Limits of Impurities

Insoluble in Ammonia			0.001	per cent.
Chloride (Cl) .			0.005	per cent.
			0.12	per cent.
Phosphate (P <sub>2</sub> O <sub>5</sub> )			0.0002	per cent.
Heavy Metals .			0.0005	per cent.
Nitrate (N2O5) .	٠		0.002	per cent.
Ammonia (NH.)			0.002	per cent.

In addition to the tests for Molybdic Acid A.R., this should conform to the following tests:—

### Solubility

1 gram should dissolve in 10 c.c. of ammonia, warming if necessary, and should not leave more than a faint trace of insoluble matter.

#### Nitrate

1 gram shaken with 10 c.c. of water and 0  $\cdot$  5 c.c. of indigo solution should remain blue on addition of 10 c.c. of sulphuric acid.

#### Ammonio

1 gram boiled with 5 c.c. of sodium hydroxide solution should not have any ammoniacal odour.

#### Assay

Treated as described in the preceding monograph not less than 99 per cent. of  $MoO_3$  should be indicated.

## α-NAPHTHOL €0.R A.R.

 $C_{10}H_2OH = 144.06$ 

#### Maximum Limits of Impurities

Alcohol-ins	olul	ble matt	ter			nil
Ash .						0.05 per cent.
Reaction						neutral
Substances	in	soluble	in	Sodi	um	
Hydroxic	le					nil

Colourless or slightly pink crystals with a characteristic odour; readily soluble in alcohol and in benzene.

### Melting Point

94°.

#### Ash

2 grams should not leave more than 1 milligram of residue on ignition.

#### Acidity

1 gram shaken with 100 c.c. of water and filtered should give a filtrate which is neutral to litmus paper.

### Naphthalene, etc.

1 gram dissolved in 2 e.e. of sodium hydroxide solution and 10 c.c. of water should form a clear and almost colourless solution free from any odour of naphthalene.

## $\beta$ -NAPHTHOL $\longrightarrow$ A.R.

 $C_{10}H_7OH = 144 \cdot 06$ 

Maximum Limits of Impurities

Alcohol-insoluble matte	er			nil
Ash				0.02 per cent.
a-Naphthol				no reaction
Substances insoluble	in	Sodi	um	
Hydroxide				nil

Colourless or faintly pink crystals with a characteristic odour; slightly soluble in water, readily soluble in alcohol.

### Melting Point

About 122°.

#### Ash

 ${\bf 5}$  grams should not leave more than 1 milligram of residue on ignition.

#### a-Naphthol

0.1 gram dissolved in 10 c.c. of boiling water, cooled and filtered, and 0.5 c.c. of ferric chloride solution added to the filtrate, should give a white precipitate, becoming brown, but not violet.

#### Naphthalene

1 gram dissolved in 2 c.c. of sodium hydroxide solution and 10 c.c. of water should form a perfectly clear solution free from any odour of naphthalene.

### α-NAPHTHYLAMINE SOH A.R.

 $C_{10}H_7NH_2 = 143 \cdot 08$ 

## Maximum Limits of Impurities

Alcol	hol-ii	nsolul	ole ma	tter		nil	
Acid	-inso	luble :	matter	٠.		nil	
Ash						0.025	per cent.

Colourless or silvery grey crystals with a characteristic odour; readily soluble in alcohol.

### Melting Point

49° to 51°.

#### Ash

2 grams moistened with sulphuric acid and ignited should not leave more than 0.5 milligram of residue.

#### Solubility

1 gram should dissolve in 15 c.c. of warm acetic acid forming a clear solution.

#### Assay

Dissolve 2·5 grams in dilute hydrochloric acid, cool and titrate against  $\rm M/2~NaNO_2$  using starch iodide paper as indicator.

1 c.c. 
$$M/2$$
 NaNO<sub>2</sub>  $\equiv 0.07154$  gram  $C_{10}H_7NH_2$ 

Not less than 99 per cent, should be indicated.

### NICKEL SULPHATE A.R.

(Cobalt and Iron free)

 $NiSO_4.6H_9O = 262.84$ 

Maximum Limits of Impurities

Chloride (Cl)			0.003	per cent.
Iron (Fe) .			0.003	per cent.
Cobalt (Co)			0.0005	par cont

An emerald green crystalline powder, readily soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of 1 c.c. of silver nitrate solution.

### Iron and Cobalt

Dissolve 5 grams in 20 c.c. of water, saturate the solution with ammonium thiocyanate and shake with 10 c.c. of a mixture of equal volumes of amyl alcohol and ether; separate and reject the aqueous layer. Wash the ethereal liquid with two separate 10 c.c. portions of a saturated solution of ammonium thiocyanate. The colour of the ethereal solution should not be deeper than that obtained by treating 0.1 milligram of ferric iron in the same manner. Remove the colour due to iron by shaking the ethereal liquid vigorously with three successive 10 c.c. portions of 40 per cent. solution of sodium thiosulphate saturated with ammonium thiocyanate. The ethereal liquid should become almost colourless.

### NITRIC ACID

 $HNO_3 = 63 \cdot 02$ 

#### Maximum Limits of Impurities

		4
	0.0001	per cent.
	0.0002	per cent.
	0.0002	per cent.
	0.00001	per cent.
		. 0.0001 . 0.0002 . 0.0005 . 0.0001

A clear, fuming liquid.

## Specific Gravity

About 1.42.

#### Residue

50 c.c. evaporated to dryness on a water-bath and ignited gently should not leave more than 0.5 milligram of residue.

#### Hydrochloric Acid

10 c.c. diluted with 50 c.c. of water should not show any opalescence on addition of silver nitrate solution.

#### Sulphuric Acid

To 50 c.c. of the acid, add 0·1 gram of sodium carbonate and evaporate to dryness; dissolve the residue in 50 c.c. of water, add barium chloride solution and allow to stand for 12 hours. No turbidity or precipitate should be formed.

#### Iodic Acid

Dilute 20 c.c. with 20 c.c. of water, add a small granule of zinc and 3 c.c. of chloroform and shake. The chloroform should not become violet.

#### Heavy Metals and Iron

20 c.c. of the acid rendered alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Calcium

The above solution should not show any turbidity on the further addition of ammonium oxalate solution.

#### Arsenic

Limit 0.1 part per million.

To 50 c.c. of the acid add 5 c.c. of sulphuric acid and evaporate down until fumes of sulphuric acid are given off; cool, add 5 c.c. of water, and again evaporate to fuming; cool, dilute with 50 c.c. of water, add 10 c.c. of stannated hydrochloric acid and test as described on page 189.

#### Assav

Titrate about 3 grams diluted with water against N/1 NaOH using methyl red as indicator.

1 e.e. N/1 NaOH  $\equiv 0.06302$  gram  $HNO_3$ 

About 70 per cent. of HNO3 should be indicated.

### NITROBENZENE A.R.

 $C_6H_5NO_2 = 123 \cdot 05$ 

A pale yellow, highly refractive liquid with a characteristic almond odour.

Specific Gravity about 1·209 Refractive Index at 20° 1·553 Freezing Point 1·509

### OXALIC ACID

 $(COOH)_2 \cdot 2H_2O = 126 \cdot 05$ 

### Maximum Limits of Impurities

Ash .			0.02 per cent.
Chloride (C	l) .		0.002 per cent.
Sulphate (SC	$O_3$ ).		0.005 per cent.
Nitrate (N <sub>2</sub> C	$O_5$ ).		0.002 per cent.
Heavy Metal			o ooi per cent.
Calcium (Ca	1) .		0.005 per cent.
Magnesium	(Mg).		o·oi per cent.

Colourless crystals, readily soluble in water and in alcoho forming clear, colourless solutions.

5 grams ignited in a platinum dish should not leave more than 1 milligram of residue.

# Hydrochloric Acid

2 grams dissolved in 50 c.c. of water and acidified with nitrie acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphuric Acid

5 grams dissolved in 150 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show any precipitate or turbidity on adding barium chloride solution and allowing to stand for 12 hours.

#### Nitric Acid

I gram dissolved in 10 c.c. of water, and 0.5 c.c. of indigo solution added, should remain blue on addition of 10 e.e. of sulphuric acid.

### Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water and rendered alkaline with ammonia should not show more than the faintest darkening on addition of 1 drop of sodium sulphide solution.

1 gram should form a clear solution in 25 c.c. of water, and should remain clear on adding ammonia and allowing to stand for six hours.

No turbidity should be produced on adding ammonium phosphate to the above solution and allowing to stand for 2 hours.

#### Assav

Dissolve 3 grams in water and titrate against N/1 NaOH using phenolphthalein as indicator. The titration should be carried out at the boiling point.

1 c.c.  $N/1 \text{ NaOH} = 0.06302 \text{ gram (COOH)}_2.2H_2O$ 

Or dissolve about 0.3 gram in water, add dilute sulphuric acid, heat to  $60^\circ$  and titrate against  $\rm N/10~KMnO_4.$ 

1 c.e. N/10 KMnO<sub>4</sub>  $\equiv$  0.006302 gram (COOH)<sub>2</sub>.2H<sub>2</sub>O

Not less than 99.8 per cent, should be indicated.

### PERCHLORIC ACID (60 per cent.)

 $HClO_4 = 100 \cdot 46$ 

### Maximum Limits of Impurities

Ash		0.02 per cent.
Hydrochloric Acid (Cl)		0.001 per cent.
Sulphuric Acid (SO <sub>3</sub> ) .		0.005 per cent.
Chloric Acid (ClO <sub>3</sub> ) .		0.001 per cent.
Heavy Metals and Iron .		0.001 per cent.
Barium (Ba)		0.002 per cent.

A clear colourless liquid, miscible with water and with alcohol forming clear, colourless solutions.

#### Specific Gravity

About 1.54.

5 c.c. evaporated to dryness and ignited gently should not leave more than I milligram of residue.

## Hydrochloric Acid

2 c.c. diluted with 20 c.c. of water should not show more than a faint opalescence on addition of silver nitrate solution and nitric acid.

### Sulphuric Acid

2 c.c. diluted with 20 c.c. of water should not show any turbidity on addition of barium chloride solution and 0.4 c.c. of hydrochloric acid.

#### Chloric Acid

5 c.c. diluted with 20 c.c. of water and 0.1 c.c. of indigo solution added should remain blue on addition of 5 c.c. of sulphurous acid (5 per cent.) and 5 c.c. of dilute sulphuric acid.

#### Heavy Metals and Iron

2 c.c. diluted with 20 c.c. of water and rendered alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Barium \*

5 c.c. diluted with 20 c.c. of water should not show any turbidity on addition of 1 c.c. of dilute sulphuric acid.

#### Acces

Titrate about 5 grams diluted with water against N/1 NaOH using phenolphthalcin as indicator.

1 c.c. N/1 NaOH = 0 · 1005 gram HClO<sub>4</sub>

Not less than 60 per cent, of IIClO<sub>4</sub> should be indicated.

### PERCHLORIC ACID

(20 per cent.)

#### Maximum Limits of Impurities

Ash		0.02 per cent.
Hydrochloric Acid (Cl)		0.001 per cent.
Sulphuric Acid (SO <sub>3</sub> ) .		0.005 per cent.
Chloric Acid (ClO <sub>3</sub> ) .		0.001 per cent.
Heavy Metals and Iron .		0.001 per cent.
Barium (Ba)		0.002 per cent.

### Specific Gravity

About 1 . 12.

This should conform to the tests in the preceding monograph.

### PETROLEUM ETHER A.R.

Maximum Limit of Impurities

Non-volatile residue . . . 0.002 per cent.

A clear, colourless, mobile, highly inflammable liquid.

#### Boiling Ranges

(1) below 40°	(5) 60° to 80°	
(2) 40° to 50°	(6) 80° to 100°	
(3) 40° to 60°	(7) 100° to 120°	3
(4) 50° to 60°	(8) Above 120	٥

#### Specific Gravity

From 0-630 to 0-780, varying with the boiling range.

#### Residue

50 c.c. evaporated on a water-bath should not leave more than 1 milligram of residue.

#### **Boiling Range**

Distil 100 c.c. from a Wurtz flask at a rate of 1 to 2 drops per second. Not less than 90 per cent. should distil between the temperatures stated.

### PETROLEUM ETHER A.R.

(Free from aromatic hydrocarbons)

#### Maximum Limits of Impurities

Non-volatile residue . . . 0 · 002 per cent. Aromatic Hydrocarbons . . 0 · 5 per cent.

Shake 10 c.c. of the sample with 30 c.c. of 98 per cent. sulphuric acid for 30 minutes. Separate the petroleum layer, wash with water and dry over calcium chloride. Mix 5 c.c. of this with 5 c.c. of freshly distilled aniline and warm until clear. Allow to cool very slowly and note the temperature, to the nearest 0·1°, at which the mixture first becomes cloudy.

the mixture first becomes cloudy.

Determine the clouding point for a mixture of the original petroleum ether and aniline in the same manner. Each degree centigrade difference between the two readings is equivalent to 1 per cent. of aromatic hydrocarbons.

Not more than 0.5 per cent. should be indicated.

Note.—If the petroleum boils below the clouding point, carry out the test with a mixture of equal parts of petroleum ether, boiling range  $100^\circ$  to  $120^\circ$  (free from aromatic hydrocarbons), and the sample.

### PHENOL A.R.

 $C_6H_5OH = 94.05$ 

### Maximum Limits of Impurities

Non-volatile matter . . . o · o · per cent.
Ash . . . . . nil
Water-insoluble matter . . nil
Ålkali-insoluble matter . . nil

Colourless hygroscopic crystals, soluble in 20 parts of water forming a clear, colourless, oily solution.

#### Freezing Point

40° to 41°.

#### Non-volatile Matter

5 grams evaporated on a water-bath should not leave more than 1 milligram of residue.

#### Ash

10 grams should leave no residue on ignition.

### Alkali-insoluble Matter

5 grams should dissolve in 15 c.c. of sodium hydroxide solution forming a clear colourless solution.

### PHENOLPHTHALEIN A.R.

 $(C_6H_4.OH)_2: C.C_6H_4.CO.O = 302.1$ 

#### Maximum Limits of Impurities

Ash					0.1	per cent.
Insoluble in Al	cohol				nil	•
Insoluble in	Sodiu	m H	ydrox	iide		
solution .			٠.		nil	
Chloride (Cl)					0.001	per cent.
Sulphate (SO.)					10.0	per cent.

An almost white, odourless powder, sparingly soluble in water. Soluble in alcohol forming a clear solution. Soluble in solutions of alkalis forming deep red, clear solutions.

#### Melting Point

Not below 254°

#### A.L

1 gram moistened with sulphuric acid and ignited should not leave more than 1 milligram of residue.

#### Chloride

Shake I gram with 20 c.c. of water and filter; the filtrate acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

Shake 1 grain with 20 c.c. of water and filter; the filtrate acidified with 1 c.c. of hydrochloric acid should not show more than a faint turbidity on addition of 1 c.c. of barium chloride solution.

### PHENYLHYDRAZINE HYDROCHLORIDE A.R.

 $C_6H_5.NH.NH_2.HCl = 144.54$ 

Maximum Limit of Impurities

Sulphated ash . . . o · 5 per cent.

White or slightly coloured crystals or crystalline powder, darkening on exposure to air and light.

#### Sulphated Ash

1 gram moistened with sulphuric acid should not leave more than 5 milligrams of residue on ignition.

#### Assay

Dissolve 1 gram in sufficient water to produce 250 c.c. Titrate 50 c.c. of this solution after adding 50 c.c. of hydrochloric acid with  $M/20~{\rm KIO_3}$ , using a globule of chloroform to indicate the disappearance of free iodine.

1 e.e.  $M/20~KIO_3 \equiv 0.007227~gram~C_6H_5.NH.NH_2.HC1$ 

 $C_6H_5.NH.NH_2.HCl+KIO_3+2HCl=C_6H_5Cl+KCl+ICl+3H_2O+N_2$ 

Not less than 98 per cent. should be indicated.

### PHLOROGLUCINOL A.R.

 $C_8H_9(OH)_3, 2H_9O\ 1:3:5=162\cdot08$ 

### Maximum Limits of Impurities

Sulphated ash			o·1 per cent.
Resorcin .			no reaction
Diresorcin .	_		no reaction

White or cream coloured crystals, readily soluble in water and in alcohol forming clear solutions.

#### Melting Point

218° to 219° (anhydrous).

#### Sulphated Ash

1 gram moistened with sulphuric acid should not leave more than 1 milligram of residue on ignition.

#### Resorcin

Heat  $0\cdot 1$  gram with  $0\cdot 1$  gram of phthalic anhydride and  $0\cdot 5$  gram of zinc chloride; cool, dissolve in water and add 1 c.c. of sodium hydroxide solution. No fluorescence should be observed.

#### Diresorcin

Boil 0·1 gram with 10 c.c. of acetic anhydride; cool and layer the solution on sulphuric acid. No violet ring should appear.

### PHOSPHOMOLYBDIC ACID

 $20\text{MoO}_3.2\text{H}_3\text{PO}_4.48\text{H}_2\text{O} = 3940 \cdot 8$ 

### Maximum Limits of Impurities

Insoluble matter		nil	
Heavy Metals and Iron .		0.001	per cent.
Calcium (Ca)		0.03	per cent.
Ammonia (NH.)		0.004	per cent.

Brilliant yellow crystals, soluble in water forming a clear, yellow solution, which should remain clear on addition of nitric acid, and on the subsequent addition of a few drops of ammonia should give a yellow precipitate.

### Heavy Metals and Iron

1 gram dissolved in 20 c.e. of water should not show more than a slight darkening on addition of excess of ammonia and 1 drop of sodium sulphide solution.

#### Calaine

I gram dissolved in 10 c.c. of water should not give any precipitate on addition of excess of ammonia and a few drops of ammonium oxalate solution.

### PHOSPHORIC ACID

 $H_3PO_4=98\!\cdot\!05$ 

### Maximum Limits of Impurities

Hydrochloric Acid (Cl)		o·ooo3 per cent.
Sulphuric Acid (SO <sub>3</sub> ) .		0.003 per cent.
Nitric Acid (N <sub>2</sub> O <sub>5</sub> ) .		0.001 per cent.
Phosphorous Acid		no reaction
Metaphosphoric Acid .		no reaction
Heavy Metals and Iron .		0.001 per cent.
Calcium and Magnesium		0.006 per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )		0.0001 per cent.
Oxygen absorbed (O).		0.002 per cent.

A colourless syrupy liquid, miscible with water and alcohol.

#### Specific Gravity

About 1.750.

#### Hydrochloric Acid

 ${f 2}$  c.c. diluted with 20 e.e. of water should not show any opalescence on addition of silver nitrate solution.

#### Sulphuric Acid

2 c.c. diluted with 50 c.c. of water should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

#### Nitric Acid

2 c.c. diluted with 10 c.c. of water and 0.5 c.c. indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Phosphorous Acid

2 c.c. warmed with 10 e.c. of water and 2 c.c. of mercuric chloride solution should not show any turbidity.

### Metaphosphoric Acid

1 c.c. diluted with water should not show any turbidity on mixing carefully with a dilute solution of albumen.

#### Heavy Metals and Iron

2 c.c. diluted with 20 c.c. of water and rendered alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Calcium and Magnesium

2 c.c. diluted with 10 c.c. of water and rendered alkaline with ammonia should not show any turbidity on addition of ammonium oxalate and heating on a water-bath for 1 hour.

#### Arsenic

Limit 1 part per million.

Test as described on page 189 using 10 grams and 10 c.c. of stannated hydrochloric acid.

#### Reducing Substances

Mix 5 c.c. with 20 c.c. of water and 0·1 c.c. of N/10 KMnO $_4$  and heat on a water-bath for 5 minutes. The solution should retain a pink colour.

#### Assay

Titrate 4 grams with 150 c.c. of water against N/1 NaOH to a blue colour using B.D.H. Universal Indicator (pH =  $9\cdot2$ ).

1 e.e. 
$$N/1 \text{ NaOH} = 0.04902 \text{ gram } H_3PO_4$$

About 89 per cent. should be indicated.

### PHOSPHOTUNGSTIC ACID

(Nitrogen free)

P<sub>9</sub>O<sub>5</sub>.24WO<sub>3</sub>.xH<sub>2</sub>O

#### Maximum Limits of Impurities

Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.003	per	cent.
Chloride (Cl)			0.01	per	cent.
Ammonia (NH <sub>3</sub> )			0.001	per	cent.

Very pale yellow crystals, soluble in water forming a clear solution with an acid reaction.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Chlorida

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### A-----

1 gram dissolved in 40 c.c. of water should not give a deeper colour on addition of 5 c.c. of sodium hydroxide solution and 2 c.c. of Nessler's reagent than is given by  $0\cdot01$  milligram of ammonia.

# PICRIC ACID A.R. (Trinitrophenol)

 $C_6H_2(NO_2)_3OH = 229 \cdot 05$ 

### Maximum Limits of Impurities

Ash		0.1	per c	ent.
Chloride (Cl) .		0.002	per c	ent.
Sulphate (SO <sub>3</sub> ) .		0.003	per c	ent.
Organic impurities		passes	test	

Pale yellow crystals, slightly soluble in water and in alcohol forming clear deep yellow solutions; completely soluble in 20 parts of benzene.

### Ash

1 gram should not leave more than 1 milligram of residue on careful ignition.

### Melting Point

122° to 123°.

#### Chloride

Dissolve 2 grams in 50 c.c. of boiling water and 1 c.c. of nitric acid, cool and filter; to the filtrate add 10 c.c. of water and 1 c.c. of silver nitrate solution. Not more than a faint opalescence should be produced.

#### Sulphate

Dissolve 2 grams in 50 c.c. of boiling water and 1 c.c. of hydrochloric acid, cool and filter; to the filtrate add 10 c.c. of water and 1 c.c. of barium chloride solution. Not more than a faint turbidity should be produced.

#### Organic Impurities

Dissolve 0·2 gram in 20 c.c. of water, add 0·5 c.c. of sodium hydroxide solution, allow to stand for 15 minutes and dilute with 20 c.c. of water. The colour of the resulting solution should not be deeper than that of a solution prepared by dissolving 0·4 gram of the same trinitrophenol in 40 c.c. of water.

#### Assay

Titrate about 1 gram dissolved in water against N/10 NaOH using phenol red as indicator.

1 c.c. N/10 NaOH  $\equiv$  0·0229 gram C<sub>6</sub>H<sub>2</sub>(NO<sub>2</sub>)<sub>3</sub>OH Not less than 99·8 per cent. should be indicated.

## POTASSIUM ALUM

 $AlK(SO_4)_2.12H_2O = 474 \cdot 37$ 

#### Maximum Limits of Impurities

Chloride (Cl)			o.ooi per cent.
Heavy Metals			0.001 per cent.
Iron (Fe)			0.001 per cent.
Ammonia (NH <sub>3</sub> )			o·ooi per cent.

Colourless crystals, soluble in water forming a clear colourless solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Heavy Metals

1 gram dissolved in 20 c.c. of water should not show more than a faint darkening on addition of 10 c.c. of hydrogen sulphide water.

#### Iron

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show more than a faint blue colour on addition of 1 c.c. of potassium ferrocyanide solution.

#### Ammonia

1 gram powdered and mixed with 1 gram of anhydrous sodium carbonate and heated strongly should not have any ammoniacal odour and should not evolve any alkaline vapours.

### POTASSIUM BICARBONATE

### $KHCO_3 = 100 \cdot 11$

### Maximum Limits of Impurities

Chloride (Cl)			0.005 per cent.
Sulphate (SO <sub>3</sub> )			0.005 per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.002 per cent.
Carbonate .			passes test
Heavy Metals and	l Iron		0.0005 per cent.
Ammonia (NH <sub>3</sub> )	•		0.003 per cent.

A white crystalline powder, soluble in water forming a clear colourless solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of 1 e.c. of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 6 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 6 hours.

1 gram dissolved in 10 e.c. of dilute sulphuric acid and 0.5 e.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Carbonate

1 gram dissolved in 100 c.c. of CO2 free cold water should not have a pH greater than 8.6 using thymol blue as indicator.

Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

1 gram heated in a dry test tube should not evolve ammonia and the vapour should not change the colour of moistened red litmus paper.

#### Assav

Titrate about 4 grams dissolved in water against N/1 H<sub>2</sub>SO<sub>4</sub> using bromo-phenol blue as indicator.

1 c.c. N/1 H<sub>2</sub>SO<sub>4</sub> = 0·10011 gram KHCO<sub>3</sub>

Not less than 99.8 per cent. should be indicated.

### POTASSIUM BISULPHATE A.R. (FUSED)

 $\mathrm{KHSO_4} = 136 \!\cdot\! 17$ 

### Maximum Limits of Impurities

Water-insoluble matter	٠.		nil		
			0.002	per ce	ent.
Nitrate $(N_2O_5)$ .			0.002		
Heavy Metals and Iron			0.001	per c	ent.
Ammonia (NH <sub>3</sub> ) .			0.0015	per c	ent.
Arsenic $(As_2O_3)$ .			0.0001		
				-	

Opaque white hygroscopic lumps, readily soluble in water forming a clear solution.

#### Chloride

1 gram dissolved in 20 c.c. of water should not show any opalescence on addition of silver nitrate solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

1 gram dissolved in 20 c.c. of water and made alkaline with ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Ammonia

Heavy Metals and Iron

1 gram together with 1 gram of sodium hydroxide (ammonia free) dissolved in 50 c.c. of water should not on addition of Nessler's reagent give a deeper colour than that given by 0.015 milligram of ammonia.

#### Arsenic

Limit 1 part per million.

Test 10 grams with 5 c.c. of stannated hydrochloric acid as described on page 189.

### Assay

Dissolve 5 grams in water and titrate against  $N/1\,$  NaOH using methyl red as indicator.

1 c.c. N/1 NaOH = 0·1362 gram KHSO<sub>4</sub>

Not less than 99 per cent. should be indicated.

### POTASSIUM BROMATE A.R.

 $KBrO_3 = 167 \cdot 02$ 

### Maximum Limits of Impurities

Bromide			no reaction
Sulphate (SO <sub>3</sub> )			0.005 per cent.

A white crystalline powder, slightly soluble in water forming a clear, colourless, neutral solution.

#### Bromide

1 gram dissolved in 20 c.c. of cold water should remain colourless on addition of 1 gram of tartaric acid.

#### Sulphate

I gram dissolved in 20 c.c. of water and acidified with hydrochloric acid should not show any turbidity on addition of barium chloride solution,

#### Assay

Dissolve 0.1 gram in water, add potassium iodide, acidify with hydrochloric acid and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> = 0.002783 gram KBrO<sub>3</sub>

Not less than 99.9 per cent, should be indicated.

### POTASSIUM BROMIDE

 $KBr = 119 \cdot 02$ 

#### Maximum Limits of Impurities

Moisture			0.5 per cent.
Free Alkali (K2CO3)			o·o35 per cent.
Chloride (Cl) .			0.25 per cent.
Bromate			no reaction
Iodide			no reaction
Sulphate (SO <sub>3</sub> ) .			
Thiocyanate (SCN)			o·oo6 per cent.
Heavy Metals and Iron	n.		0.001 per cent.

White crystals, readily soluble in water forming a clear solution.

#### Moisture

5 grams of the powdered salt should not lose more than 25 milligrams on drying at 130°.

#### Free Alkali

5 grams dissolved in 100 c.c. of water should not require more than 0.25 c.c. of N/10  $\rm H_2SO_4$  to produce a greenish-yellow colour with B.D.H. Universal Indicator.

#### Chloride

Dissolve 2 grams in 75 e.e. of water, add 25 e.e. of nitric acid, boil gently and bubble air through until the liberated bromine is removed and the liquid is colourless. Cool and titrate against  $\rm N/10~AgNO_3$ .

Not more than 1.5 c.c. should be required.

#### Brom

Shake 1 gram with 10 c.c. of water, 2 c.c. of chloroform and 1 c.c. of dilute sulphuric acid; the chloroform should not be coloured yellow.

#### Iodide

1 gram dissolved in 10 c.c. of water and shaken with 5 drops of ferric chloride solution and starch solution should not develop a blue colour.

#### Sulphate

I gram dissolved in 20 e.e. of water and acidified with 0.4 e.e. of hydrochloric acid should not show any turbidity on adding barium chloride solution and standing for 6 hours.

#### Thiocyanate

I gram dissolved in 10 c.c. of water and acidified with hydrochloric acid should not show any red colour on addition of 1 drop of ferric chloride solution.

#### Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water should not show any darkening on addition of 1 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Assay

Dissolve about 0.5 gram, previously dried at  $130^\circ,$  in water and titrate against N/10  ${\rm AgNO_3}$  by Volhard's method. Correct the titration figure for the amount of chloride present.

### 1 c.c. $N/10 \text{ AgNO}_3 \equiv 0.01190 \text{ gram KBr}$

Not less than 99 per cent. should be indicated.

# POTASSIUM CARBONATE A.R.

 $K_2CO_3 = 138 \cdot 2$ 

## Maximum Limits of Impurities

Water-insoluble 1	matte	r.		nil	
Chloride (Cl)				0.003	per cent.
Sulphate ( $SO_3$ )				0.005	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )				0.002	per cent.
Heavy Metals and	l Iron	1.		0.001	per cent.
Arsenic (As <sub>0</sub> O <sub>0</sub> )	_		_	0.0001	ner cent

A white granular hygroscopic powder, readily soluble in water forming a clear, colourless solution; insoluble in alcohol.

#### Chlorida

2 grams dissolved in 50 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 8 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and standing for 6 hours.

#### Nitrate

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

## Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water should not show any darkening on addition of 1 drop of sodium sulphide solution.

## Arsenic

Limit 1 part per million.

Dissolve 10 grams in 40 c.c. of water, add 25 c.c. of brominated hydrochloric acid followed by a few drops of stannous chloride solution and test as described on page 189.

#### Assay

Dissolve 3 grams in water and titrate against  $\rm N/1~H_2SO_4$  using brome-phenol blue as indicator.

1 c.c. 
$$N/1 H_2SO_4 \equiv 0.0691 \text{ gram } K_2SO_3$$

Not less than 99 per cent, should be indicated.

# POTASSIUM CHLORATE A.R.

## $KClO_3 = 122 \cdot 56$

## Maximum Limits of Impurities

Reaction .			neutral	
			0.0004	per cent.
+ +				per cent.
				per cent.
Calcium (Ca)			-	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )			0.00002	per cent.

Colourless crystals, soluble in water forming a clear, colourless solution neutral to litmus.

### Chloride

1 gram dissolved in 20 c.e. of water should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

I gram dissolved in 20 c.c. of water should not show any turbidity on adding barium chloride solution and allowing to stand for 6 hours.

#### Heavy Metale

5 grams dissolved in 50 c.e. of hot water should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Calcium

1 gram dissolved in 20 c.c. of water should not show any turbidity on addition of ammonia and ammonium oxalate solution.

### Arsenic

Limit 0.2 part per million.

Mix 5 grams with 20 c.c. of water and 22 c.c. of hydrochloric acid, warm gently until chlorine ceases to be evolved, cool, add 20 c.c. of water and a few drops of stannous chloride solution, and test as described on page 189.

# POTASSIUM CHLORIDE A.R.

#### KCl = 74.56

## Maximum Limits of Impurities

Moisture .				0.2	per cent.
Sulphate (SO <sub>3</sub> )				0.003	per cent.
Nitrate $(N_2O_5)$				0.002	per cent.
Heavy Metals and	l Iro	n.		0.0005	per cent.
Calcium and Mag	gnesi	um		0.01	per cent.
Barium (Ba)				0.005	per cent.

A white crystalline powder, readily soluble in water forming a clear, colourless solution.

#### Neutrality

 $^5$  grams dissolved in neutral distilled water should give a solution neutral to B.D.H. Universal Indicator (greenish-yellow) or which should not require more than  $0\cdot05$  c.c. of N/10 NaOH or  $N/10\,H_2\mathrm{SO}_4$  to produce neutrality.

#### Moisture

5 grams should not lose more than 10 milligrams on drying at 130.

#### Sulphate

5 grams dissolved in 100 c.c. of water should not show any turbidity on adding barium chloride solution and allowing to stand for 2 hours.

#### Milanda

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

### Heavy Metals and Iron

 $2~{\rm grams}$  dissolved in 50 c.c. of water should not show any darkening on addition of 1 drop of sodium sulphide solution.

### Calcium and Magnesium

2 grams dissolved in 50 c.c. of water should not show any turbidity on addition of ammonia, ammonium oxalate and ammonium phosphate solutions.

## Barium

2 grams dissolved in 20 c.c. of water should not show any turbidity on adding dilute sulphuric acid, and allowing to stand for 2 hours.

#### Acons

Titrate 0·3 gram of the previously dried salt dissolved in water against N/10 AgNO<sub>3</sub>.

1 c.c. N/10 AgNO<sub>3</sub> = 0 · 007456 gram KCl

Not less than 99.9 per cent. should be indicated.

## POTASSIUM CHROMATE A.R.

 $K_0CrO_4 = 194 \cdot 21$ 

## Maximum Limits of Impurities

Chloride (Cl)			0.001	per	cent.
Sulphate (SO <sub>3</sub> )			0.02	per	cent.
Calcium (Ca)			0.002	per	cent.
Aluminium (Al)			0.003	per	cent.

Lemon yellow crystals, readily soluble in water forming a clear yellow solution which should not be more than faintly alkaline to litmus.

## Chloride

2 grams dissolved in 100 c.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

2 grams dissolved in 100 c.c. of water and acidified with 3 c.c. of hydrochloric acid should not show any turbidity on adding 2 c.c. of barium chloride solution and allowing to stand for 2 hours.

### Aluminium and Calcium

2 grams dissolved in 50 c.c. of water should not show any turbidity on addition of ammonia and ammonium oxalate solution.

#### Assay

Dissolve 0.3 gram in water, add potassium iodide solution and hydrochloric acid and titrate the liberated iodine against  $N/10~\mathrm{Na_2S_2O_3}$  using starch solution as indicator.

1 c.c. N/10 
$$Na_2S_2O_3 = 0.006474$$
 gram  $K_2CrO_4$ 

Not less than 99 per cent. should be indicated.

## POTASSIUM CITRATE

 $C_3H_4$ . OH (COOK)<sub>3</sub>.  $H_9O = 324 \cdot 36$ 

## Maximum Limits of Impurities

Granular crystals or a white crystalline powder, readily soluble in water forming a clear colourless solution.

#### Reaction

5 grams boiled with 50 c.c. of water require for neutralisation to phenolphthalein not more than 0.5 c.c. of either N/10 NaOH or  $H_2SO_4$ .

### Chloride

Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

1 gram dissolved in 20 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show more than a faint turbidity or precipitate on addition of 1 c.c. of barium chloride solution.

#### Heavy Metals and Iron

 $1\ gram\ dissolved$  in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

## Organic Impurities

2 grams heated in a boiling water-bath for 1 hour with 10 e.e. of sulphuric acid should not acquire more than a pale yellow colour.

Reducing Substances

10 grams dissolved in 50 c.c. of hot water and boiled for 5 minutes with 4 grams of anhydrous sodium carbonate and 10 c.c. of copper sulphate solution should remain clear.

#### Assay

Char about 4 grams in a platinum dish, boil the residue with 50 c.c. of  $\rm N/1~H_2SO_4$  and 100 c.c. of water ; filter, wash and titrate the excess of acid against N/1 NaOH.

1 c.e. N/1 
$$H_2SO_4 \equiv 0.1081$$
 gram  $K_3C_6H_5O_7.H_2O$ 

Not less than 99 per cent. should be indicated.

## POTASSIUM CYANIDE A.R.

### $KCN = 65 \cdot 11$

### Maximum Limits of Impurities

Insoluble in 60 per cer	nil		
Chloride (Cl) .			0.5 per cent.
Sulphate (SO <sub>3</sub> ) .			o oi per cent.
Ferrocyanide (Fe(CN	) <sub>6</sub> )		0.004 per cent.
Thiocyanate (SCN)			o or per cent.
Sulphide			no reaction
Heavy Metals .			o oot per cent.
Sodium			no vellow flame

White lumps or crystals with a characteristic odour of hydrocyanic acid; readily soluble in water forming a clear, colourless solution, strongly alkaline to litmus.

#### Solubility

Should dissolve completely in hot 60 per cent. alcohol.

#### Chloride

Dissolve 1 gram in 50 c.c. of water, add 25 c.c. of formaldehyde solution, 5 c.c. of nitric acid and an excess of N/10 silver nitrate. Titrate the excess of silver against N/10 NH<sub>4</sub>SCN using iron alum as indicator. Not more than 0.5 per cent. of chloride, calculated as Cl. should be indicated.

#### Sulphate

1 gram dissolved in 50 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

### Ferrocyanide and Thiocyanate

1 gram dissolved in 20 c.c. of water and acidified with hydrochloric acid should not give either a blue or a red colour on addition of 1 drop of ferric chloride solution.

#### Sulphide

1 gram dissolved in 20 e.e. of water should not show any darkening on addition of 1 e.e. of ammonia and 1 drop of lead ammonio-citrate solution.

## Heavy Metals

1 gram dissolved in 20 c.c. of water should not darken in colour on addition of 10 c.c. of hydrogen sulphide water, nor on further addition of 2 c.c. of hydrochloric acid.

### Sodium

Moistened with hydrochloric acid and heated in a flame on platinum wire, no yellow flame should be produced.

#### Assay

Dissolve 0.5 gram in 25 c.c. of water, add 10 c.c. of ammonia and 1 drop of potassium iodide solution and titrate against  $N/10~AgNO_3$  until a faint permanent turbidity appears.

1 c.c. N/10 AgNO<sub>3</sub> = 0.01302 gram KCN

Not less than 96 per cent, should be indicated.

# POTASSIUM DICHROMATE A.R.

 $K_9Cr_9O_7 = 294 \cdot 22$ 

## Maximum Limits of Impurities

Moisture .			0.05 per cent.
Chloride (Cl)			0.001 per cent.
Sulphate (SO <sub>3</sub> )			o or per cent.
Aluminium (Al)			0.003 per cent.
Calcium (Ca)			0.005 per cent.
Sodium .			no vellow flame

Red crystals or a deep orange-red crystalline powder, soluble in water forming a clear, orange-red solution.

## Moisture

 $10~{\rm grams}$  dried at  $120^{\circ}$  should not lose more than 5 milligrams in weight.

## Chloride

2 grams dissolved in 50 e.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

2 grams dissolved in 50 c.c. of water and acidified with 3 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 6 hours.

#### Aluminium and Calcium

2 grams dissolved in 50 c.c. of water should not show any turbidity on addition of excess of ammonia or on further addition of ammonium oxalate solution.

### Sodium

Moistened with hydrochloric acid and heated in a flame on platinum wire, no yellow flame should be produced.

#### 4ssa3

Dissolve 0.2 gram in water, add potassium iodide solution and hydrochloric acid and titrate the liberated iodine against  $N/10~Na_2S_2O_3$  using starch solution as indicator.

1 e.e. 
$$N/10 \text{ Na}_2S_2O_3 \equiv 0.004904 \text{ gram } K_2Cr_2O_7$$

Not less than 99.9 per cent. should be indicated.

# POTASSIUM DIHYDROGEN PHOSPHATE A.R.

 $KH_2PO_4 = 136 \cdot 14$ 

## Maximum Limits of Impurities

Insoluble matter			nil
Reaction of aqueous sol	ution		pH 4.5
			o·I per cent.
Chloride (Cl)			0.001 per cent.
Sulphate (SO <sub>3</sub> )			o or per cent.
Heavy Metals and Iron .			0.001 per cent.

Colourless crystals, readily soluble in water forming a clear colourless solution.  ${}^{\bullet}$ 

#### Reaction

An aqueous solution containing 9 grams per litre should have a pH value of 4.5 (bromo-cresol green).

#### Moisture

5 grams dried at  $100^{\circ}$  should not lose more than 5 milligrams in weight.

### Chloride

I gram dissolved in 20 e.e. of water and acidified with 1 c.e. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 1 hour.

## Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water should not show more than a slight darkening on addition of 5 c.c. of ammonia and 1 drop of sodium sulphide solution.

#### Assay

Dissolve 5 grams in 500 c.c. of water and titrate to pH 9.2 against N/1 NaOH using B.D.H. Universal Indicator (blue).

Not less than 99.5 per cent. should be indicated.

# POTASSIUM FERRICYANIDE A.R.

 $K_3 Fe(CN)_6 = 329 \cdot 19$ 

Maximum Limits of Impurities

Sulphate  $(SO_3)$  . . . o o o per cent. Ferrocyanide  $(Fe(CN)_6)$  . o o o per cent.

Ruby red crystals, readily soluble in water forming a clear solution.

## Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

### Ferrocyanide

1 gram rapidly washed with water and then dissolved in 100 c.c. of water should not show any blue colour on addition of 1 drop of ferric chloride solution.

#### Assay

Dissolve 1 gram in 50 c.c. of water, add 5 grams of potassium iodide, 3 grams of zinc sulphate and 20 c.c. of hydrochloric acid, and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 
$$\equiv$$
 0 · 03292 gram K<sub>3</sub>Fe(CN)<sub>6</sub>

Not less than 99 per cent. should be indicated.

# POTASSIUM FERROCYANIDE A.R.

 $K_4 Fe(CN)_6.3 H_2 O = 422.33$ 

## Maximum Limits of Impurities

Chloride (Cl) . . . 0 · 003 per cent. Sulphate (SO<sub>3</sub>) . . . 0 · 005 per cent.

Pale yellow granular crystals, readily soluble in water forming a clear yellow solution.

#### Chloride

Dissolve 1 gram in 10 c.c. of water, precipitate with a slight excess of copper sulphate solution and filter. The filtrate, acidified with nitric acid, should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Assay

Dissolve 2 grams in 50 c.c. of water, acidify with sulphuric acid and titrate against  $N/10~KMnO_4$ .

1 e.e.  $KMnO_4 \equiv 0.04223 \text{ gram } K_4Fe(CN)_6.3H_2O$ 

Not less than 99 per cent. should be indicated.

# POTASSIUM HYDROGEN PHTHALATE

 $COOH.C_6H_4.COOK = 204.14$ 

Maximum Limits of Impurities

Insoluble matter			nil	
Moisture .				per cent.
Chloride (Cl)			0.001	per cent.
Sulphate (SO <sub>3</sub> )			0.01	per cent.
Heavy Metals			0.001	per cent.

A white crystalline powder, slightly soluble in water forming a clear colourless solution.

#### Moisture

5 grams dried at  $100^{\circ}$  should not lose more than 5 milligrams in weight.

#### Chloride

1 gram dissolved in 30 e.c. of water and acidified with nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 30 c.c. of water and acidified with hydrochloric acid should not show any turbidity on addition of barium chloride solution.

## Heavy Metals

1 gram dissolved in 30 c.c. of water and rendered alkaline with ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Assay

Titrate  $5 \cdot 1035$  grams dissolved in hot water against N/1 NaOH using thymol blue as indicator.

1 e.e. N/1 NaOH 
$$\equiv 0.2041$$
 gram COOH.  $C_6H_4$ . COOK

Not less than 99.9 nor more than 100.1 per cent, should be indicated.

# POTASSIUM HYDROGEN TARTRATE A.R.



Maximum Limits of Impurities

Moisture			0.1	per cent.
Chloride (Cl) .			0.001	per cent.
Sulphate $(SO_3)$ .			0.002	per cent.
Heavy Metals and Iro	n.		0.0002	per cent.

A white crystalline powder, sparingly soluble in water.

#### Moietu

5 grams dried at  $100^{\circ}$  should not lose more than 5 milligrams in weight.

1 gram dissolved in 10 c.c. of water and 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Salabate

1 gram dissolved in 10 c.c. of water and 1 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

## Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water and 5 c.c. of ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Assay

Titrate 9.407 grams diffused in 200 c.c. of hot water against N/1 NaOH using phenolphthalein as indicator and boiling well towards the end of the titration.

1 c.c. N/1 NaOH = 0·1881 gram KHC<sub>4</sub>H<sub>4</sub>O<sub>6</sub>

Not less than 99.9 per cent. should be indicated.

# POTASSIUM HYDROXIDE A.R.

## KOH == 56·11

## Maximum Limits of Impurities

Chloride (Cl) • .			0.01	per cent.
Sulphate (SO <sub>3</sub> ) .			0.005	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> ) .			0.002	per cent.
Heavy Metals and Iron	n.		100.0	per cent.
Alumina (Al) .			0.01	per cent.
Silica (SiÒ <sub>2</sub> )			0.01	per cent.
Ammonia (NH <sub>3</sub> )			0.001	per cent.
Arsenic (As <sub>0</sub> O <sub>3</sub> ) .			0.0001	per cent.
Carbonate (K <sub>2</sub> CO <sub>3</sub> )			2'5	per cent.

White deliquescent sticks or pellets, very soluble in water forming a clear alkaline solution. Soluble in alcohol leaving only a small quantity undissolved (carbonate).

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

## Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 10 c.c. of hydrochloric acid should not show any turbidity on adding 2 c.c. of barium chloride solution and allowing to stand for 2 hours.

#### Nitrate

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

# Heavy Metals and Iron

 $1\ gram\ dissolved$  in  $50\ c.c.$  of water should not show any darkening on addition of  $1\ drop$  of sodium sulphide solution.

#### Alumina

5 grams dissolved in 50 c.c. of water and 5 c.c. of glacial acetic acid added, followed by 20 c.c. of ammonia, should not show more than a faint turbidity on heating on a water-bath for 2 hours.

### Silica

Dissolve 5 grams in 5 c.c. of water, add 10 c.c. of hydrochloric acid, evaporate to dryness and bake. The residue, dissolved in very dilute hydrochloric acid, should form a clear solution free from any insoluble matter.

### Ammonia

1 gram dissolved in 50 c.c. of ammonia-free water should not give a deeper colour on addition of 2 c.c. of Nessler's reagent than that given by 0.01 milligram of ammonia.

## Arsenic

Limit 1 part per million.

Dissolve 5 grams in 40 c.c. of water, add 20 c.c. of brominated hydrochloric acid and a few drops of stannous chloride solution, and test as described on page 189.

#### Assay

Dissolve 3 grams in 200 c.c. of  $\mathrm{CO}_2$  free cold water and titrate against N/1  $\mathrm{H}_2\mathrm{SO}_4$  using phenolphthalein (note amount required =a), then add bromo-phenol blue and continue the titration against N/1  $\mathrm{H}_2\mathrm{SO}_4$  (amount required =b); calculate (a-b) to KOH and 2b to  $\mathrm{K}_2\mathrm{CO}_3$ .

1 c.c. N/1 
$$H_2SO_4 \equiv 0.05611$$
 gram KOH  $\equiv 0.0691$  gram  $K_2CO_3$ 

Not less than 85 per cent. of KOH nor more than  $2\cdot 5$  per cent. of  $K_2CO_3$  should be indicated.

# POTASSIUM IODATE A.R.

 $KIO_3 = 214 \cdot 03$ 

## Maximum Limits of Impurities

Reaction . . . . neutral to litmus Iodide . . . . . no reaction

A white crystalline powder, soluble in water forming a clear colourless solution neutral to litmus.

#### Indida

1 gram shaken with 20 c.c. of water, 1 c.c. of  $N/10~H_2SO_4$  and 2 c.c. of chloroform should not impart a violet colour to the chloroform.

#### Assay

Dissolve 0.15 gram in 30 c.c. of water, add potassium iodide solution and 5 c.c. of hydrochloric acid, and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. N/10 Na<sub>0</sub>S<sub>0</sub>O<sub>2</sub>=0.003567 gram KIO<sub>3</sub>

Not less than 99.9 per cent. should be indicated.

# POTASSIUM IODIDE

KI = 166.03

## Maximum Limits of Impurities

Free Alkali (K <sub>2</sub> CO <sub>3</sub> )		o·o4 per cent.
Moisture		o·5 per cent.
Chloride and Bromide		passes test
Sulphate $(SO_3)$ .		o·oi per cent.
Iodate		no reaction
Heavy Metals and Iron		o ooi per cent.

White crystals, readily soluble in water forming a clear colour less solution; soluble in alcohol.

#### Free Alkal

 $1~\rm gram$  dissolved in 20 c.c. of  $\rm CO_2$  free water should not show more than a faint pink colour on addition of 1 drop of phenolphthalein solution.

#### Moisture

5 grams powdered and dried at  $120^{\circ}$  should not lose more than 25 milligrams in weight.

#### Chloride and Bromide

Dissolve 0.5 gram in 10 c.c. of water, add 5 c.c. of ammonia and 31 c.c. of  $N/10 \text{ AgNO}_3$ , shake well and filter; the filtrate should not show more than a faint opalescence on acidifying with nitric acid.

#### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.4 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and standing for 1 hour.

#### Iodate

1 gram dissolved in 20 c.c. of water should not show any blue colour on addition of tartarie acid solution and starch solution.

## Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

## Assay

Dissolve 0.5 gram of the previously dried salt in 10 c.c. of water, add 35 c.c. of hydrochloric acid and titrate with  $M/20~{\rm KIO_3}$ , shaking vigorously until the dark brown solution which is formed becomes light brown; add 5 c.c. of chloroform and continue the titration with vigorous shaking until the chloroform globule becomes colourless and the supernatant liquid is clear yellow.

Not less than 99.5 per cent, should be indicated.

# POTASSIUM METABISULPHITE

$$K_2S_2O_5 = 222 \cdot 32$$

## Maximum Limits of Impurities

Chloride (Cl)			0.002 p	er	cent.
Heavy Metals			0.001 b	er	cent.
Arsenic (As <sub>0</sub> O <sub>0</sub> )			0.00002 pe	er	cent.

Colourless crystals or a white powder, soluble in water forming a clear solution.

1 gram dissolved in 20 c.c. of water and acidified with 2 c.c. of nitric acid should not show more than a slight opalescence on addition of 1 c.c. of silver nitrate solution.

#### Heavy Metals

1 gram dissolved in 40 e.c. of water should not show more than a faint darkening on addition of ammonia and 1 drop of sodium sulphide solution.

# Arsenic

Limit 0.2 parts per million.

Treat 5 grams with 3 grams of potassium chlorate, 10 c.c. of water and 20 c.c. of hydrochloric acid, and when the reaction has ceased, boil gently to remove chlorine; add 40 c.c. of water and a few drops of stannous chloride solution and test as described on page 189.

#### Assay

Dissolve about 0.25 gram in 50 c.c. of N/10 I and titrate the excess of iodine against N/10  $\rm Na_2S_2O_3$ .

1 e.c. of N/10 I  $\equiv$  0.005559 gram of  $K_2S_2O_5$ 

Not less than 98 per cent. should be indicated.

# POTASSIUM NITRATE A.R.

 $\text{KNO}_3 = 101 \cdot 11$ 

## Maximum Limits of Impurities

Reaction .				neutral
Chloride (Cl)				o oor per cent.
Sulphate (SO <sub>3</sub> )				0.005 per cent.
Nitrite (N <sub>2</sub> O <sub>5</sub> )				o · ooo1 per cent.
Heavy Metals an	d Iroi	n.		0.0005 per cent.

Colourless crystals, readily soluble in water forming a clear colourless solution neutral to litmus.

## Chloride

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

### Sulphate

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 6 hours.

#### Nitrite

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of sulphuric acid should not show any yellow colour on addition of *m*-phenylene-diamine sulphate solution.

### Heavy Metals and Iron

2 grams dissolved in 50 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

# POTASSIUM OXALATE (NEUTRAL)

 $(\mathrm{COOK})_2.\mathrm{H}_2\mathrm{O} = 184 \!\cdot\! 22$ 

# Maximum Limits of Impurities

Reaction .					not greater than pH 8.5
					nor less than pH 7·0
Chloride (Cl)					0.0005 per cent.
Sulphate (SO					0.005 per cent.
Heavy Metals	and Iro	n.	•	•	o ooi per cent.

Colourless crystals, readily soluble in water forming a clear colourless solution, the pH of which is between 7.0 and 8.5.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 2 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

# Sulphate

1 gram dissolved in 20 c.c. of water and acidified with  $2\cdot 5$  e.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 2 hours.

### Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water and rendered alkaline with ammonia should not show any darkening on addition of 1 drop of sodium sulphide solution.

# POTASSIUM PERMANGANATE

 $KMnO_4 = 158 \cdot 03$ 

# Maximum Limits of Impurities

Chloride (Cl)			0.002 per cent.
Sulphate $(SO_3)$			0.006 per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.005 per cent.

Brilliant black crystals with a metallic lustre, free from any blue or violet iridescence; soluble in water forming a deep purple solution free from any insoluble matter.

Dissolve 2 grams in 50 c.c. of water, heat on a water-bath and add alcohol little by little until the permanganate is reduced and the supernatant liquid is colourless.

Filter and use the filtrate for the following tests:-

#### Chloride

10 c.c. of the filtrate acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

10 c.c. of the filtrate acidified with hydrochloric acid should not show more than a faint turbidity on adding barium chloride solution and allowing to stand for 1 hour.

#### Nitrate

CUL 11 (CI)

10 e.e. of the filtrate with 0.5 c.c. of indigo solution should remain blue on addition of 10 c.c. of sulphuric acid.

# POTASSIUM PERSULPHATE A.R.

 $\mathrm{KSO_4} = 135 \!\cdot\! 16$ 

### Maximum Limits of Impurities

Chioride (Ci)	•	•	•	0.003	per cent.
Ammonia (NH <sub>3</sub> )				0.001	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )				0.0001	per cent.
Manganese (Mn)				0.0005	per cent.

A white crystalline powder, soluble in water forming a clear colourless solution.

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of 1 c.c. of silver nitrate solution.

#### Ammonia

1 gram dissolved in 50 c.c. of water should not show a deeper colour on addition of 2 c.c. of Nessler's reagent than is shown by • 0 · 01 milligram of NH<sub>o</sub>.

### Arsenic

Limit 1 part per million.

Mix 10 grams with 20 c.c. of hydrochloric acid and boil gently to remove free chlorine, dilute with water, add a few drops of stannous chloride solution and test as described on page 189.

#### Manganese

1 gram boiled with 10 c.c. of water, 10 c.c. of dilute nitric acid and 1 c.c. of  $N/10~AgNO_3$  should not show any pink colour.

# POTASSIUM SULPHATE A.R.

$$K_2SO_4 = 174 \cdot 26$$

## Maximum Limits of Impurities

Reaction .			not less than pH 6.0
			nor greater than pH 8.5
Chloride (Cl)			0.0005 per cent.
Nitrate $(N_2O_5)$	•.		0.002 per cent.
Heavy Metals and			0.001 per cent.
Ammonia (NH <sub>3</sub> )			o·oo15 per cent.

Opaque white crystals, soluble in water forming a clear colourless solution, the pH of which is between  $6\cdot0$  and  $8\cdot5$ .

## Chloride

1 gram dissolved in 20 e.e. of water and acidified with 0.5 e.e. of nitric acid should not show any opalescence on addition of silver nitrate solution.

### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

### Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### A-monio

1 gram dissolved in 50 c.c. of water should not give a deeper colour on addition of Nessler's reagent than that given by  $0\cdot015$  milligram of ammonia.

## POTASSIUM TETROXALATE A.R.

$$KH_3(C_2O_4)_2.2H_2O = 254.15$$

## Maximum Limits of Impurities

Chloride (Cl)				0.001	per	cent.
Sulphate (SO <sub>3</sub> )				0.01	per	cent.
Calcium (Ca)				0.01	per	cent.
Heavy Metals an	d Iro	m.		0.001	per	cent.

Colourless transparent crystals, slightly soluble in water forming a clear colourless solution.

#### Chloride

1 gram dissolved in 50 c.c. of water acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 50 c.c. of water acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

## Calcium, Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water and 5 c.c. of aminonia should form a clear solution, and should not show any darkening on addition of 1 drop of sodium sulphide solution.

#### Assav

Titrate 4 grams dissolved in hot water against N/1 NaOH using phenolphthalein as indicator.

1 c.e. N/1 NaOH = 
$$0.08472$$
 gram KH<sub>3</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>2</sub>, 2H<sub>2</sub>O

Titrate 0.8 gram dissolved in hot water and acidified with dilute sulphuric acid against N/10 KMnO<sub>4</sub>.

1 c.c. N/10 KMnO<sub>4</sub> 
$$\equiv$$
 0.006354 gram KH<sub>3</sub>(C<sub>2</sub>O<sub>4</sub>)<sub>2</sub>.2H<sub>2</sub>O

Not less than 99.9 per cent. should be indicated.

## POTASSIUM THIOCYANATE A.R.

 $KSCN = 97 \cdot 17$ 

## Maximum Limits of Impurities

Alcohol-insoluble matt	er		nil
Chloride (Cl) .			0.004 per cent.
Sulphate $(SO_3)$ .			o·oi per cent.
Heavy Metals .			o · ooı per cent.
Iron (Fe)			0.0002 per cent.
Ammonia (NH <sub>3</sub> ) .			0.002 per cent.
Isothiocyanate (NCS)			no reaction

Colourless deliquescent crystals, readily soluble in water forming a clear colourless solution. Completely soluble in 10 parts of hot alcohol.

#### Chloride

Dissolve 1 gram with 1 gram of ammonium nitrate in 30 e.c. of hydrogen peroxide (20 volumes), add 1 gram of sodium hydroxide and rotate the flask gently from time to time until a vigorous reaction commences. When this has abated, add a further 30 e.c. of hydrogen peroxide and boil for 2 minutes; cool, acidify with nitric acid and add 1 e.c. of silver nitrate solution. Not more than a faint opalescence should be produced.

## Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

## Heavy Metals

1 gram dissolved in 20 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Iron

1 gram dissolved in 10 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any pink colour.

#### Ammonia

1 gram dissolved in 5 c.c. of water and boiled with sodium hydroxide solution should not have any ammoniacal odour.

#### Isothiocyanate

5 grams dissolved in 25 c.c. of water and 3 c.c. of silver nitrate solution added, and the precipitate redissolved by thorough shaking, should not darken in colour on warming with 20 c.c. of ammonia for 15 minutes.

# PYRIDINE A.R.

 $C_5H_5N=79\!\cdot\!05$ 

## Maximum Limits of Impurities

Residue		0.01	per cent.
Chloride (Cl) .		0.001	per cent.
Oxygen absorbed (O)		0.0006	per cent.
Copper (Cu) .		0.0004	per cent.

A clear colourless liquid with a characteristic odour. Miscible with water forming a clear, colourless solution.

### Specific Gravity

0.988 to 0.989.

## **Boiling Range**

114° to 117°.

#### Residue

 $10~\rm c.c.$  evaporated to dryness and gently ignited should not leave more than 1 milligram of residue.

#### Water

Should form a clear solution with an equal volume of carbon disulphide.

### Chloride

1 c.c. dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of 1 c.c. of silver nitrate solution.

## Reducing Substances

5 c.c. mixed with 0.05 c.c. of N/10 KMnO  $_4$  should retain a pink colour for 1 hour.

## Copper

Dissolve 5 c.c. in 10 c.c. of water and 5 c.c. of glacial acetic acid, add 5 c.c. of potassium thiocyanate and 5 c.c. of chloroform, shake vigorously and allow to separate. The chloroform layer should not be coloured yellow or green.

## Assav

Dissolve about 3 grams in water and titrate against N/1 HCl using bromo-phenol blue as indicator.

1 c.c. N/1 HCl  $\equiv$  0 · 07905 gram C<sub>5</sub>H<sub>5</sub>N

Not less than 99 per cent. should be indicated.

# QUINHYDRONE A.R.

 $C_6H_4O_2.C_6H_4(OH)_2 = 218.08$ 

Maximum Limits of Impurities

Ash . . . . . . o · r per cent.

Insoluble matter . . . nil

A dark green crystalline powder, completely soluble in hot alcohol.

## Melting Point

170° to 173°.

#### A.L

1 gram should not leave more than 1 milligram of residue on ignition.

#### Assar

Dissolve about 0·4 gram in 10 c.c. of warm alcohol, cool, add 25 c.c. of water, 15 c.c. of hydrochloric acid and 3 grams of potassium iodide and titrate the liberated iodine immediately with  $N/10~Na_9S_9O_3$ .

1 c.c. N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> 
$$\equiv$$
 0 · 0109 gram C<sub>12</sub>H<sub>10</sub>O<sub>4</sub>

Not less than 99 per cent. should be indicated.

Dissolve about 0.4 gram in a mixture of 40 c.c. of N/10 I, 20 c.c. of water, 1 c.c. of acetic acid and 5 grams of sodium acetate and titrate the excess of iodine against N/10  $\rm Na_2S_2^2O_3$ .

1 c.c. N/10 I 
$$\equiv$$
 0.0109 gram  $C_{12}H_{10}O_4$ 

Not less than 99 per cent. should be indicated.

# RESORCINOL A.R.

 $C_6H_4(OII)_2 = 110.05$ 

Maximum Limits of Impurities

Small colourless granular crystals, very soluble in water and in alcohol forming clear colourless solutions. On exposure to air and light, or to ammonia, it acquires a pink colour.

## Melting Point

110° to 112°.

#### Ach

5 grams should not leave more than  $0\cdot 5$  milligram of residue on ignition.

### Acid

1 gram dissolved in 10 c.c. of water should not require more than 1 drop of N/10 NaOH to produce a blue colour with bromo-cresol green.

### Diresorcinol and Phenol

1 gram should dissolve in 10 c.c. of water, forming a clear solution, and there should be no phenolic odour on warming.

# SALICYLIC ACID

 $C_6H_4$ .OH.COOH 1:2 = 138.05

## Maximum Limits of Impurities

Ash		0.02	per cent.
Chloride (Cl)		0.001	per cent.
Heavy Metals and Iron		0.0005	per cent.

Colourless needle crystals, slightly soluble in cold water, readily soluble in alcohol and organic solvents.

## Melting Point

157° to 159°.

#### Ash

5 grams should not leave more than 2 5 milligrams of residue on ignition.

#### Hydrochloric Acid

I gram dissolved in 50 c.c. of hot water and acidified with 1 c.c. of mitric acid should not show any opalescence on addition of silver nitrate solution,

### Heavy Metals and Iron

5 grams dissolved in 10 c.c. of ammonia and 40 c.c. of water should form a clear and almost colourless solution and should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

## Organic Impurities

0.5 gram should dissolve in 10 c.c. of sulphuric acid with not more than a slight colour.

#### Assav

Titrate 4 grams dissolved in alcohol against  $N/1\,$  NaOH using phenol red as indicator.

1 c.c. N/1 NaOH = 0 · 1380 gram C<sub>6</sub>H<sub>4</sub> · OH · COOH

Not less than 99.9 per cent, should be indicated.

# SILVER NITRATE A.R.

 $AgNO_3 = 169 \cdot 89$ 

Maximum Limits of Impurities

Alcohol-insoluble matter . . nil
Copper, Bismuth and Lead . . "no reaction

Colourless crystals, readily soluble in water forming a clear colourless solution. Soluble in alcohol.

## Copper, Bismuth and Lead

1 gram dissolved in 5 c.c. of water should form a clear, colourless solution on addition of 10 c.c. of ammonia.

#### Acen

Dissolve 0.5 gram in 50 c.c. of water, acidify with nitric acid and precipitate with potassium iodide. Filter off on a Gooch crucible, wash, ignite gently and weigh the AgI.

$$\frac{AgNO_3}{AgI} \equiv 0.7235$$

Not less than 99.9 per cent. should be indicated,

## SODIUM ACETATE

 $CH_3COONa.3H_9O = 136.07$ 

## Maximum Limits of Impurities

Chloride (Cl) .		0.0005	per cent.
Sulphate ( $SO_3$ ) .		0.005	per cent.
Heavy Metals and Iron		0.001	per cent.
Calcium (Ca)		0.01	per cent.

Colourless crystals, readily soluble in water and in alcohol forming clear colourless solutions, which should not be more than faintly alkaline to phenolphthalein.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

#### Heavy Metals and Iron

 $1\ gram\ dissolved$  in 20 e.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Calcium

1 gram dissolved in 20 c.c. of water should not show any turbidity on addition of ammonium oxalate solution.

# SODIUM ACETATE A.R. (FUSED)

 $CH_3COONa = 82 \cdot 02$ 

White or pale grey crystalline masses, readily soluble in water.

#### Assay

Moisten 1 gram with sulphuric acid, ignite and weigh the resulting Na $_2$ SO  $_4$ 

$$\frac{2CH_3COONa}{Na_2SO_4} \equiv 1 \cdot 155$$

Not less than 99 per cent, should be indicated.

## SODIUM BICARBONATE .A.R.

 $NaHCO_{3} = 84.00$ 

## Maximum Limits of Impurities

Carbonate .			trace
Chloride (CI)			0.005 per cent.
Sulphate (SO <sub>4</sub> )			0.005 per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )			0.002 per cent.
Heavy Metals and	Iron		0.0005 per cente
Ammonia (NH <sub>3</sub> )			0.003 per cent.

A white soft powder, soluble in about 12 parts of cold water forming a clear solution.

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 2 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 8 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding 1 c.c. of barium chloride solution and allowing to stand for 6 hours.

#### Niti

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c of indigo solution added should remain blue on addition of 10 c.c of sulphuric acid.

#### Carbonate

1 gram dissolved in 100 c.c. of water should not have a pH greater than  $8\cdot 6$ , using thymol blue as indicator.

#### Heavy Metals and Iron

 $2~{\rm grams}$  dissolved in 40 c.c. of water should not show any darkening  $\,$  on addition of ammonia and 1 drop of sodium sulphide solution.

## Ammonia

1 gram heated in a dry test tube should not evolve ammonia and the vapour should not change the colour of moistened red litmus paper.

#### Assay

Titrate 4 grams dissolved in water against  $\rm N/1~H_2SO_4$ , using bromo-phenol blue as indicator.

1 c.c. 
$$N/1 H_2SO_4 \equiv 0.084 \text{ gram NaHCO}_3$$

Not less than 99.5 per cent. should be indicated.

# SODIUM BISMUTHATE

 $NaBiO_3 = 280 \cdot 0$ 

Maximum Limits of Impurities

Manganese (Mn) . . . . 0.0005 per cent. Chloride (Cl) . . . . 0.001 per cent.

A yellow or brown amorphous powder, insoluble in water.

#### Manganese

2 grams boiled with 12 c.c. of nitric acid and 20 c.c. of water should produce an almost colourless solution free from any pink tint.

#### Chloride

Boil 1 gram with 25 c.c. of water for 10 minutes, dilute to 50 c.c. with water, and filter. To the filtrate add 1 c.c. of nitric acid and 1 c.c. of silver nitrate solution. No opalescence should be produced.

#### Assay

Treat 0.5 gram with 20 c.c. of water, 5 grams of potassium iodide and 20 c.e. of hydrochloric acid. Allow to stand for 30 minutes, and titrate the liberated iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c.  $N/10 \text{ Na}_2S_2O_3 \equiv 0.0140 \text{ gram NaBiO}_3$ 

Not less than 85 per cent, should be indicated,

# SODIUM BISULPHATE A.R. (FUSED)

 $NaHSO_4 = 120 \cdot 06$ 

Maximum Limits of Impurities

Opaque white hygroscopic lumps, readily soluble in water forming a clear acid solution.

1 gram dissolved in 20 c.c. of water should not show any opalescence on addition of silver nitrate solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Heavy Metals and Iron

 $1\,\mathrm{gram}$  dissolved in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Ammonia

1 gram, together with 1 gram of sodium hydroxide (ammonia free), dissolved in 50 c.c. of water should not produce a deeper colour on addition of 2 c.c. of Nessler's reagent than that given by 0.015 milligram of ammonia.

#### Arsenic

Limit 1 part per million.

Test as described on page 189 using 10 grams with 10 c.c. of stannated hydrochloric acid.

#### Assay

Dissolve 5 grams in water and titrate against  $N/1\,$  NaOII, using methyl red as indicator.

1 c.c. N/1 NaOH  $\equiv$  0·1201 gram NaHSO<sub>4</sub>

Not less than 99 per cent, should be indicated.

# SODIUM BORATE A.R.

(Borax)

 $Na_{2}B_{4}O_{7}.10H_{2}O = 381.43$ 

## Maximum Limits of Impurities

Chloride (Cl)				0.001	per cent.
Sulphate (SO <sub>3</sub> )				0.005	per cent.
Carbonate .	•			no reac	tion
Heavy Metals an	d Iro	n.		0.001	per cent.
					per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )				0.0002	per cent.

Transparent crystals or a white crystalline powder, soluble in water forming a clear colourless solution.

1 gram dissolved in 30 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

## Sulphate

2 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

#### Carhonste

A cold saturated solution should not effervesce on addition of hydrochloric acid.

### Heavy Metals and Iron

1 gram dissolved in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

#### Calcium

I gram dissolved in 30 c.c. of water should not show any turbidity on addition of ammonium oxalate solution.

#### Arsenic

Limit 5 parts per million.

Test as described on page 189 using 2 grams with 4 grams of citric acid and 12 c.c. of stannated hydrochloric acid.

#### Assay

Dissolve 5 grams in water and titrate against N/1  $H_2SO_4$ , using brome-phenol blue as indicator.

1 c.c. N/1  $H_2SO_4 \equiv 0.1907$  gram  $Na_2B_4O_7.10H_2O$ Not less than 99 per cent. should be indicated.

# SODIUM CARBONATE (ANHYDROUS) A.R.

 $Na_2CO_3 = 105 \cdot 99$ 

Maximum Limits of Impurities

Water-insoluble matter		nil		
Chloride (Cl) .		0.003	per	cent.
Sulphate (SO <sub>3</sub> ) .		0.005	per	cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> ) .		0.002	per	cent.
Heavy Metals and Iron		0.001	per	cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> ) .		0.00002	per	cent.
Ammonia		0000	•	

A white powder, readily soluble in water forming a clear colourless solution.

2 grams dissolved in 50 c.c. of water and acidified with 4 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 10 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 12 hours.

## Nitrate

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

## Heavy Metals and Iron

 $2~{
m grams}$  dissolved in  $40~{
m c.c.}$  of water should not show any darkening on addition of ammonia and  $1~{
m drop}$  of sodium sulphide solution.

#### Ammonia

5 grams dissolved in 50 c.c. of water should not show any yellow colour on addition of 2 c.c. of Nessler's reagent.

#### Arconic

Limit 0.2 part per million.

Dissolve 5 grams in 16 c.c. of brominated hydrochloric acid and 50 c.c. of water, add a few drops of stannous chloride solution and test as described on page 189.

## Assay

Titrate 2.650 grams dissolved in water against N/1  $\rm\,H_2SO_4$  at the boiling point, using phenolphthalein as indicator.

1 c.c.  $N/1 H_2SO_4 \equiv 0.053 \text{ gram Na}_0CO_3$ 

Not less than 99.9 per cent, should be indicated.

## SODIUM CHLORIDE

 $NaCl = 58 \cdot 45$ 

## Maximum Limits of Impurities

Reaction .				neutral
Moisture .				o·i per cent.
Sulphate (SO				0.001 per cent.
Nitrate (N2O				o·ooi per cent.
Heavy Metals		n.		0.001 per cent.
Barium (Ba)				0.002 per cent.
Calcium (Ca)				0.005 per cent.
Magnesium (I	Mg).			0.005 per cent.

White crystals, readily soluble in water forming a clear, colourless, neutral solution.

#### Moisture

5 grams should not lose more than 5 milligrams on drying at 130°.

### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding 1 c.c. of barium chloride solution and allowing to stand for 24 hours.

### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain, blue on addition of 10 c.c. of sulphuric acid.

## Heavy Metals and Iron

 $2~\rm grams$  dissolved in 40 e.e. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

### Alkaline Earths

2 grams dissolved in 20 c.c. of water should not show any turbidity on adding dilute sulphuric acid and allowing to stand for 2 hours. 2 grams dissolved in 20 c.c. of water should not show any turbidity on adding ammonia, ammonium oxalate and sodium phosphate solutions and allowing to stand for 2 hours.

#### Assa

Titrate 0·2923 gram of the freshly ignited salt dissolved in water against N/10  ${\rm AgNO_3}.$ 

1 c.c.  $N/10 \text{ AgNO}_3 \equiv 0.005845 \text{ gram NaCl}$ 

Not less than 99.9 per cent, should be indicated.

## SODIUM CITRATE

 $C_3H_4$ .OII (COONa)<sub>3</sub>,2 $H_2O = 294.06$ 

## Maximum Limits of Impurities

Granular crystals or a white crystalline powder, readily soluble in water forming a clear colourless solution.

#### Reaction

5 grams boiled with 50 c.c. of water require for neutralisation to phenolphthalein not more than 0.5 c.c. of either N/10 NaOH or N/10 H<sub>2</sub>SO<sub>4</sub>.

## Chloride

1 gram dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show more than a faint turbidity or precipitate on addition of 1 c.c. of barium chloride solution.

## Heavy Metals and Iron

 $1~\rm gram~dissolved$  in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

## Organic Impurities

2 grams heated in a boiling water-bath for 1 hour with 10 c.c. of sulphuric acid should not acquire more than a pale yellow colour.

#### Reducing Substances

10 grams dissolved in 50 c.c. of hot water and boiled for 5 minutes with 4 grams of anhydrous sodium carbonate and 10 c.c. of copper sulphate solution should remain clear.

### Assay

Char about 4 grams in a platinum dish, boil the residue with 50 e.e. of N/1  $H_2SO_4$  and 100 e.e. of water; filter, wash, and titrate the excess of acid against N/1 NaOH.

1 c.c.  $N/1 H_2SO_4 \equiv 0.09802 \text{ gram } Na_3C_6H_5O_7.2H_2O$ 

Not less than 99 per cent, should be indicated.

# SODIUM COBALTINITRITE A.R.

 $Na_3Co(NO_2)_6 = 403.98$ 

## Maximum Limits of Impurities

Chloride (Cl)	•		0.002	per	cent.
Sulphate (SO <sub>3</sub> )			0.01	per	cent.
Iron (Fe)			0.001	per	cent.

A deep orange-coloured powder, readily soluble in water forming a deep orange-red solution.

#### Chloride

1 gram dissolved in 40 c.c. of water, acidified with 1 c.c. of nitric acid, warmed on a water-bath with 10 c.c. of hydrogen peroxide (20 volumes) until decomposed, and cooled, should not show more than a faint opalescence on addition of 1 c.c. of silver nitrate solution.

#### Sulphate

1 gram dissolved in 40 c.c. of water, acidified with 1 c.c. of hydrochloric acid, warmed on a water-bath with 10 c.c. of hydrogen peroxide (20 volumes) until decomposed, and cooled, should not show more than a faint turbidity on addition of 1 c.c. of barium chloride solution.

## Iron

Dissolve 1 gram in 40 c.c. of water, acidify with 1 c.c. of hydrochloric acid, warm on a water-bath with 10 c.c of hydrogen peroxide (20 volumes) until decomposed, and cool. Add 10 c.c. of ammonium chloride solution and an excess of ammonia and filter through a Buchner funnel of 5 5 c.in. diameter; no brown residue should be left on the filter paper.

## Sensitivity

Dissolve 3 grams in 10 c.c. of water and add to a mixture of 1 milligram of potassium chloride dissolved in 5 c.c. of water and 2 c.c. of acetic acid, and allow to stand for 1 hour. A distinct precipitate should be formed.

# SODIUM HYDROXIDE A.R.

## NaOH = 40.00

## Maximum Limits of Impurities

Carbonate (Na <sub>2</sub> CO <sub>3</sub>	) .		2.5	per cent.
Chloride (Cl) .			0.01	per cent.
Sulphate (SO <sub>3</sub> ) .			0.005	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> ) .			0.002	per cent.
Heavy Metals and Ir	on.		0.001	per cent.
Alumina (Al) .			0.01	per cent.
Silica (SiO <sub>2</sub> ) .			0.01	per cent.
Ammonia (NH <sub>3</sub> ).			0.001	per cent.
Arsenic (As <sub>2</sub> O <sub>2</sub> ).			0.0001	per cent.

White deliquescent sticks or pellets, very soluble in water forming a clear alkaline solution. Soluble in alcohol leaving only a small quantity undissolved (carbonate).

#### Chloride

1 gram dissolved in 20 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Sulphate

5 grams dissolved in 100 c.c. of water and acidified with 13 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 2 hours.

#### Nitrate

1 gram dissolved in 10 c.c. of dilute sulphuric acid and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

### Heavy Metals and Iron

1 gram dissolved in 50 c.c. of water should not show any darkening on addition of 1 drop of sodium sulphide solution.

## Alumina

5 grams dissolved in 50 c.c. of water and 8 c.c. of glacial acetic acid added, followed by 20 c.c. of ammonia, should not show more than a faint turbidity on heating on a water-bath for two hours.

#### Silica

Dissolve 5 grams in 5 c.c. of water, add 15 c.c. of hydrochloric acid, evaporate to dryness and bake. The residue, dissolved in very dilute hydrochloric acid, should form a clear solution free from any insoluble matter.

### Ammonia

1 gram dissolved in 50 c.c. of ammonia free water should not show a deeper colour on addition of 2 c.c. of Nessler's reagent than that shown by 0.01 milligram of ammonia.

### Arsenic

Limit 1 part per million.

Dissolve 5 grams in 40 c.c. of water and 22 c.c. of brominated hydrochloric acid, add a few drops of stannous chloride solution and test as described on page 189.

### Assay

Dissolve 3 grams in 200 c.c. of  $\mathrm{CO}_2$  free cold water and titrate against N/1  $\mathrm{H}_2\mathrm{SO}_4$  using phenolphthalein (note amount required = a), then add bromo-phenol blue and continue the titration against N/1  $\mathrm{H}_2\mathrm{SO}_4$  (amount required = b). Calculate (a-b) to NaOH and 2b to Na $_2\mathrm{CO}_3$ .

1 c.e. N/1 
$$H_2SO_4 \equiv 0.0400$$
 gram NaOH  $\equiv 0.053$  gram  $Na_2CO_3$ 

Not less than 95 per cent. of NaOH nor more than  $2\cdot 5$  per cent. of Na $_2\text{CO}_3$  should be indicated.

# SODIUM NITRATE

 $NaNO_3 = 85.00$ 

## Maximum Limits of Impurities

Reaction		neutral	
Chloride (Cl)		0.006	per cent.
Sulphate (SO <sub>3</sub> )		0.005	per cent.
Phosphate $(P_2O_5)$		0.001	per cent.
Iodate $(I_2O_5)$		0.002	per cent.
Nitrite $(\tilde{N}_2\tilde{O}_3)$		0.0002	per cent.
Heavy Metals and Iron .		0.001	per cent.

Colourless crystals, readily soluble in water forming a clear, colourless, neutral solution.

## Chloride

25 grams dissolved in water should not require more than 0.4 c.c. of N/10 AgNO<sub>3</sub> to produce a red colour with potassium chromate indicator.

### Sulphate

2 grams dissolved in 40 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on adding 1 c.c. of barium chloride solution and allowing to stand for 6 hours.

### Phosphate

Dissolve 5 grams in 35 c.c. of water, add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B, and allow to stand for 5 minutes. No blue colour should be produced.

### Iodate and Nitrite

1 gram dissolved in 10 c.c. of water and acidified with 1 c.c. of dilute sulphuric acid should not show any immediate blue colour on addition of 1 c.c. of starch solution and 1 c.c. of cadmium iodide solution.

### Heavy Metals and Iron

I gram dissolved in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

# SODIUM NITRITE A.R.

 $NaNO_2 = 69 \cdot 00$ 

### Maximum Limits of Impurities

Chloride (Cl) .			0.005 per cent.
Sulphate ( $SO_3$ ) .			0.01 per cent.
Heavy Metals and Iron	١.		0.001 per cent.
Potassium (K)			0.02 per cent.

Pale yellow hygroscopic crystals, readily soluble in water forming a clear solution.

### Chloride

I gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

I gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of nitric acid should not show any turbidity on addition of barium chloride solution.

### Heavy Metals and Iron

1 gram dissolved in 40 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

### Potassium

Dissolve 2·5 grams in 10 c.c. of water, add 3 c.c. of 10 per cent. cobalt nitrate solution and 2 c.c. of acetic acid, and allow to stand for 1 hour. No precipitate nor turbidity should be produced.

#### Assav :

Dissolve 1 gram in sufficient water to produce 250 c c., and with this solution in a burette, titrate 50 c.c. of N/10 KMnO<sub>4</sub> acidified with dilute sulphuric acid.

1 c.e. N/10 KMnO<sub>4</sub>  $\equiv$  0.003450 gram NaNO<sub>2</sub>

Not less than 98 per cent, should be indicated.

# SODIUM NITROPRUSSIDE A.R.

 $Na_{9}Fe(CN)_{5}NO.2H_{9}O = 297.91$ 

# Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )			0.005 per cent.
Ferricyanide			no reaction
Ferrocyanide			no reaction

Transparent ruby red crystals, readily soluble in water forming a clear red solution.

### Sulphate

1 gram dissolved in 10 c.c. of water and acidified with 0.2 c.c. of hydrochloric acid should not show any turbidity on addition of 1 c.c. of barium chloride solution.

### Ferricyanide and Ferrocyanide

1 gram dissolved in 10 e.c. of water should not show any blue colour on addition of ferrous sulphate solution or of ferric chloride solution.

### SODIUM OXALATE A.R.

(COONa)2 = 133.99

### Maximum Limits of Impurities

				•	
Moisture					o·I per cent.
Reaction	•				not less than pH 8
					nor greater than pH 9
Chloride (			•		0.002 per cent,
Sulphate (	$5O_3$	•	•		o·oɪ per cent.
Heavy Me	tals at	nd Iro	n.	•	0.001 per cent.

A white crystalline powder, slightly soluble in water forming a clear colourless solution.

#### Moisture

 $10~{\rm grams}$  should not lose more than  $10~{\rm milligrams}$  on drying at  $105^\circ.$ 

### Neutrality

1 gram dissolved in 50 c.c. of  ${\rm CO_2}$  free cold water should give a greenish-blue colour with B.D.H. Universal Indicator.

### Chloride

1 gram dissolved in 30 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

### Sulphate

I gram dissolved in 30 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

### Heavy Metals and Iron

1 gram dissolved in 30 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

### . Assay

Char 3 grams in a platinum dish, boil the residue with 100 c.c. of water and 50 c.c. of N/1  $H_2SO_4$ , filter and wash with hot water. Titrate the excess of acid in the filtrate and washings against N/1 NaOH using phenolphthalein as indicator.

### 1 c.c. $N/1 H_2SO_4 \equiv 0.067 \text{ gram (COONa)}_2$

Titrate 0.335 gram dissolved in hot water and acidified with dilute sulphuric acid against  $N/10~KMnO_4$ .

1 c.c.  $N/10 \text{ KMnO}_4 \equiv 0.0067 \text{ gram (COONa)}_2$ 

Not less than 99.9 per cent. should be indicated.

# SODIUM PEROXIDE A.R.

 $Na_2O_2 = 77 \cdot 99$ 

### Maximum Limits of Impurities

Chloride (Cl) .			o·oo3 per cent.
Sulphate (SO <sub>3</sub> ) .		•	0.005 per cent.
Phosphate (P <sub>2</sub> O <sub>5</sub> )			0.005 per cent.
Heavy Metals and Iron	ı.		0.001 per cent.

A slightly yellowish, hygroscopic powder, readily soluble in water with evolution of oxygen and forming a clear solution.

#### Chloride

3 grams dissolved in 100 c.c. of water and acidified with 3 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

5 grams dissolved in 100 e.c. of water and acidified with 8 c.c. of hydrochloric acid should not show any turbidity or precipitate on adding barium chloride solution and allowing to stand for 12 hours.

### Phosphate

Dissolve 1 gram in 25 c.c. of water and boil the solution until the volume is reduced to 10 c.c., cool, add 15 c.c. of dilute sulphuric acid and 10 c.c. of water, and again cool, then add 10 c.c. of phosphate reagent A and 5 c.c. of phosphate reagent B. No blue colour should be produced in 5 minutes.

### Heavy Metals and Iron

1 gram dissolved in 20 c.c. of water should not darken in colour after boiling and adding 1 drop of sodium sulphide solution.

### Assay

Weigh out about 0.2 gram in a dry flask, add a mixture of 10 c.c. of potassium iodide solution and 10 c.c. of dilute hydrochloric acid, and titrate the liberated iodine against N/10 Na<sub>S</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. N/10 
$$Na_2S_2O_3 \equiv 0.0039$$
 gram  $Na_2O_2$ 

Not less than 85 per cent, should be indicated.

# SODIUM PHOSPHATE A.R.

### $Na_2HPO_4.12H_2O = 358 \cdot 21$

### Maximum Limits of Impurities

Chloride (Cl)			0.005 per cent.
Sulphate (SO <sub>3</sub> )			o·oɪ per cent.
Carbonate .			no reaction
Heavy Metals and	Iron		o.ooo5 per cent.
Arsenic (As <sub>2</sub> O <sub>2</sub> )			0.0005 per cent.

Colourless crystals, readily soluble in water forming a clear colourless solution.

### Chloride

2 grams dissolved in 40 c.c. of water and acidified with 2 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Sulphate

2 grams dissolved in 20 c.c. of water and acidified with 2 c.c. of hydrochloric acid should not show any turbidity on adding barium chloride solution and allowing to stand for 2 hours.

### Carbonate

5 grams dissolved in 50 c.c. of boiling water should not effervesee on addition of 3 c.c. of hydrochloric acid.

### Heavy Metals and Iron

2 grams dissolved in 40 c.c. of water should not show any darkening on addition of 1 drop of sodium sulphide solution.

### Arsenic

Limit 5 parts per million.

Test as described on page 189 using 2 grams and 10 c.c. of stannated hydrochloric acid.

# SODIUM PHOSPHATE (ANHYDROUS) A.R.

 $Na_2HPO_4 = 142 \cdot 02$ 

# Maximum Limits of Impurities

Moisture .			0.1	per cent.
Reaction .			pH 9·2	per centi
Chloride (Cl)				per cent.
Sulphate (SO <sub>3</sub> )			0.025	per cent.
Carbonate .			nil	• .
Heavy Metals			0.0012	per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )			0.0012	per cent.

A white powder, readily soluble in water forming a clear colourless solution.

Sodium phosphate anhydrous should conform to the foregoing tests for sodium phosphate, proportionately less amounts being taken for the tests.

### Moisture

 $10~{\rm grams}$  should not lose more than  $10~{\rm milligrams}$  on drying at  $100^{\circ}.$ 

### Reaction

2 grams dissolved in 200 c.c. of  $\mathrm{CO}_2$  free water should have a pH value of 9·2 (blue colour with B.D.H. Universal Indicator), and after adding 5 c.c. of N/1  $\mathrm{H}_2\mathrm{SO}_4$  and boiling for 5 minutes it should require 5 c.c. of N/1 NaOH to bring back the reaction to pH 9·2.

# SODIUM POTASSIUM TARTRATE

 $NaKC_4H_4O_6.4H_2O = 282 \cdot 19$ 

Maximum Limits of Impurities

Free Alkali		nil	
Acidity (KHC <sub>4</sub> H <sub>4</sub> O <sub>6</sub> )		0.19	per cent.
Chloride (Cl) .		0.001	per cent.
Sulphate (SO <sub>3</sub> ) .		0.01	per cent.
Heavy Metals and Iron		0.0005	per cent.

Colourless crystals or a white crystalline powder, readily soluble in water forming a clear colourless solution.

(Continued overleaf)

#### Neutrality

1 gram dissolved in 10 c.c. of CO $_2$  free water should not be alkaline to phenolphthalein and the addition of 0·1 c.c. of N/10 NaOH should produce a pink colour.

#### Chloride

 $1\,$  gram dissolved in 20 c.c. of water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 1 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

### Heavy Metals and Iron

5 grams dissolved in 40 c.c. of water should not show more than a faint darkening on addition of ammonia and 1 drop of sodium sulphide solution.

### Assay

Char 5 grams in a platinum dish, boil the residue with 50 c.c. of N/1  $H_2\mathrm{SO}_4$  and 100 c.c. of water; filter, wash and titrate the excess of acid against N/1 NaOH.

1 e.c. N/1 
$$H_2SO_4 \equiv 0.1411$$
 gram  $NaKC_4H_4O_6.4II_2O$ 

Not less than 99 per cent. should be indicated.

## SODIUM SULPHATE

## $\rm Na_2SO_4.10H_2O = 322 \!\cdot\! 21$

### Maximum Limits of Impurities

Chloride (Cl) .		0.0005 per cent.
Nitrate $(N_2O_5)$ .		0.001 per cent.
Nitrite $(N_2O_3)$ .		0.0002 per cent.
Heavy Metals .		0.0002 per cent.
Iron (Fe)		0.0002 per cent.
Zinc(Zn)		0.0005 per cent.
		0.0002 per cent.
Oxidising substances		no reaction
Reducing substances		no reaction

Colourless efflorescent crystals, very soluble in water forming a clear, colourless, neutral solution.

#### Chloride

5 grams dissolved in 50 c.c. of water and acidified with 1 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

#### Nitrate

2 grams dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

#### Nitrite

5 grams dissolved in 50 c.c. of water should not show any yellow or orange colour on addition of 1 c.c. of sulphuric acid and 2 c.c. of m-phenylene-diamine sulphate solution.

#### Heavy Metals and Iron

5 grams dissolved in 40 c.c. of water should not darken in colour on addition of ammonia and 1 drop of sodium sulphide solution.

### Iron and Zinc

2 grams dissolved in 50 c.c. of water should not show any opalescence or more than a faint blue colour on adding 1 c.c. of hydrochloric acid and 1 c.c. of potassium ferrocyanide solution and allowing to stand for 1 hour.

### Arsenic

Limit 2 parts per million.

Test as described on page 189 using 5 grams and 10 c.c. of stannated hydrochloric acid.

### Oxidising and Reducing Substances

5 grams dissolved in 50 c.c. of water should remain colourless on addition of an acidified, colourless solution of potassium iodide and starch and should become blue on further addition of 1 drep of N/10~I.

## SODIUM SULPHATE (ANHYDROUS)

### $Na_{2}SO_{4} = 142 \cdot 05$

### Maximum Limits of Impurities

Moisture		o·5 per cent.
Chloride (Cl) .		0.001 per cent.
		0.002 per cent.
		0.0004 per cent
Heavy Metals .		0.0004 per cent.
Iron (Fe)		0.0004 per cent.
Zinc (Zn)		o ooi per cent.
Arsenic $(As_2O_3)$ .		0.0004 per cent.
Oxidising substances		no reaction
Reducing substances		no reaction

A white powder, readily soluble in water forming a clear, colourless, neutral solution.

Sodium sulphate anhydrous should conform to the foregoing tests for sodium sulphate, proportionately less amounts being taken for the tests.

### Moisture

10 grams should not lose more than 50 milligrams on gentle ignition.

# SODIUM SULPHIDE A.R.

 $Na_2S. 9H_2O = 240 \cdot 19$ 

### Maximum Limits of Impurities

Insoluble matter . . . nil
Other Sulphur compounds . . passes test

White or colourless deliquescent crystals, readily soluble in twice its weight of water forming a clear, alkaline solution which should not deposit a sediment on standing.

### Other Sulphur Compounds

1 gram dissolved in 10 c.c. of water should not show more than a slight milkiness on addition of 2 c.c. of hydrochloric acid.

### Assay

Dissolve I gram in 50 c.c. of water and titrate with M/10 zinc ammonium chloride solution using nickel sulphate as external indicator.

1 c.c. M/10 zinc ammonium chloride  $\equiv 0.02402$  gram  $Na_2S.9H_9O$ 

Not less than 95 per cent. should be indicated.

### SODIUM SULPHITE A.R.

 $Na_2SO_3.7H_2O = 252 \cdot 16$ 

Maximum Limits of Impurities

Chloride (Cl) .		0.005	per	cent
Heavy Metals and Iron		0.001	per	cent.
Arsenic (As <sub>2</sub> O <sub>2</sub> ) .		0.00002	per	cent

Colourless efflorescent crystals, readily soluble in water forming a clear colourless solution with an alkaline reaction.

### Chloride

1 gram dissolved in 10 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Heavy Metals and Iron

1 gram dissolved in 10 c.c. of water should not show any darkening on addition of ammonia and 1 drop of sodium sulphide solution.

### Arsenic

Treat 5 grams with 2 grams of potassium chlorate, 10 c.c. of water and 20 c.c. of hydrochloric acid, and when the reaction has ceased, boil gently to remove chlorine; add 40 c.c. of water and a few drops of stannous chloride solution and test as described on page 189.

### Assay

Add about 0.5 gram to 50 c.c. of N/10 I, and when dissolved add 2 grams of sodium bicarbonate and titrate the excess of iodine against N/10 Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>.

1 c.c. 
$$N/10 I \equiv 0.01261 \text{ gram } Na_2SO_3.7H_2O$$

Not less than 96 per cent, should be indicated.

### SODIUM THIOSULPHATE A.R.

 $Na_2S_2O_3.5H_2O = 248\cdot19$ 

### Maximum Limits of Impurities

Reaction					neutral or faintly alkaline
Sulphide					 none
Sulphate ar	id Si	ılphite	e (SO	3).	o·oɪ per cent.
Calcium (C	Ca)	٠.			0.005 per cent.

Colourless crystals, readily soluble in water forming a clear solution which should not be more than faintly alkaline to phenolphthalein.

### Sulphide

I gram dissolved in 20 c.c. of water should not show any darkening on addition of 1 drop of copper sulphate solution.

### Sulphite and Sulphate

1 gram dissolved in 10 c.c. of water should not show any turbidity on addition of a slight excess of N/10 iodine and a few drops of barium chloride solution.

1 gram dissolved in 10 c.c. of water should not show any turbidity on addition of ammonia and ammonium oxalate solution.

#### Assay

Dissolve 1 gram in water and titrate against N/10 I.

I c.c.  $N/10 I \equiv 0.02482 \text{ gram } Na_2S_2O_3.5H_2O$ 

Not less than 99 per cent. should be indicated.

# SODIUM TUNGSTATE

 $Na_2WO_4.2H_2O = 330.02$ 

Maximum Limits of Impurities

Insoluble matter			nil
Chloride (Cl)			0.003 per cent.
Sulphate (SO <sub>3</sub> )			o·oi per cent.
Nitrate (N <sub>o</sub> O <sub>c</sub> )			0.002 per cent.

Colourless crystals or a white crystalline powder, readily soluble in water forming a clear, colourless solution, the pH of which should be between 8.0 and 9.0.

#### Chloride

1 gram dissolved in 20 c.e. of hot water, acidified with nitric acid and filtered, should not show more than a faint opalescence on addition of silver nitrate solution.

I gram dissolved in 20 c.c. of hot water, acidified with hydrochloric acid and filtered, should not show any turbidity on addition or barium chloride solution.

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

1 gram should not lose more than 0.12 gram on gentle ignition.

### Assay

Dissolve 1 gram in 10 e.e. of water, add 10 e.e. of hydrochloric acid, evaporate to dryness and heat the residue at  $120^\circ$  for one hour; cool, add 20 c.c. of 20 per cent. nitric acid and again evaporate and heat at 120°; repeat the evaporation with nitric acid and subsequent heating twice. Digest the residue with 20 c.c. of ammonium nitrate solution acidified with nitric acid, filter off, wash with dilute nitric acid, dry, ignite, and weigh the  $\mathrm{WO}_3$ . Not less than 0.69 gram of  $\mathrm{WO}_3$  should be obtained.

# SOLUBLE STARCH A.R.

### Maximum Limits of Impurities

Reaction					pH 6.0 to 8.0
Moisture					15 per cent.
Chloride (	Cl)				o·oī per cent.
Reducing	uhst:	ances :	s De	xtrose.	0:25 per cent.

A soft white powder, miscible with but insoluble in cold water, soluble in hot water forming a slightly opalescent limpid solution, which after cooling should give a deep blue colour on addition of 1 drop of N/10 iodine.

### Neutrality

1 gram mixed with 20 c.c. of cold water should have a pH value between 6.0 and 8.0.

(Continued overleaf)

#### Maisture

5 grams should not lose more than 0.75 gram on drying at 100°.

#### Chloride

1 gram shaken with 20 c.c. of cold water and filtered should not show more than a faint opalescence on addition of silver nitrate solution.

### Copper Reducing Substances

10 c.c. of a 2 per cent, solution in hot water should not reduce more than 0.1 c.c. of Fehling's solution.

## STANNOUS CHLORIDE A.R.

 $SnCl_2 \cdot 2H_2O = 225 \cdot 64$ 

## Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )			0.01	per	cent.
Other Metals			0.02	per	cent.
Arsenic (As <sub>a</sub> O <sub>a</sub> )	_		0.0001	ner	cent.

Colourless hygroscopic crystals, very soluble in water forming a clear, colourless solution.

### Sulphate

1 gram dissolved in 50 c.c. of water and acidified with hydrochloric acid, should not show any turbidity on addition of barium chloride solution.

### Other Metals

Dissolve 2 grams in 100 c.c. of water and 0.5 c.c. of hydrochloric acid, remove the tin by means of hydrogen sulphide and filter. The filtrate, evaporated to dryness and ignited, should not leave more than 1 milligram of residue.

### Arsenic

Limit 1 part per million.

Dissolve 5 grams in 20 c.c. of 20 per cent. hydrochloric acid and distil 15 c.c. Test the distillate as described on page 189.

#### Assay

Dissolve 0.5 gram in 50 c.c. of water acidified with hydrochloric acid, add 5 grams of sodium potassium tartrate and a slight excess of sodium bicarbonate and titrate against N/10~I.

1 c.c. N/10 I = 0.01128 gram SnCl<sub>2</sub>.2H<sub>2</sub>O

# SUCCINIC ACID .A.R.

 $(CH_2COOH)_2 = 118.05$ 

### Maximum Limits of Impurities

Ash			0.02	per	cent.
Chloride (Cl)			0.003	per	cent.
Sulphate (SO <sub>3</sub> )			0.005	per	cent.

White crystals, readily soluble in water forming a clear colourless solution.

### Ash

1 gram leaves on ignition not more than 0.5 milligram of ash.

#### Chloride

1 gram dissolved in 20 c.c. of water should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

1 gram dissolved in 20 c.c. of water should not show any turbidity on addition of barium chloride solution.

#### Assav

Titrate about 2 grams dissolved in water against N/1 NaOH using phenolphthalein as indicator.

1 e.c. N/I NaOH  $\equiv$  0.05902 gram (CH<sub>2</sub>COOH)<sub>2</sub>

Not less than 99 per cent. should be indicated.

# SUCROSE A.R.

 $C_{12}H_{22}O_{11} = 342 \cdot 17$ 

# Maximum Limits of Impurities

Insoluble matter			nil
Ash			0.02 per cent.
Moisture .			o·r per cent.
011 11 (01)			o · ooı per cent.
Sulphate (SO <sub>3</sub> )			0.005 per cent.
Reducing Sugars			no immediate reaction
80			(Continued overleaf)

Colourless crystals or a white crystalline powder, very soluble in water forming a clear, colourless, syrupy solution.

#### A.h

10 grams should not leave more than 2 milligrams of residue on ignition.

#### Maistare

5 grams should not lose more than 5 milligrams on drying at  $100^{\circ}$  for one hour.

### Chloride

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of nitric acid should not show any opalescence on addition of silver nitrate solution.

### Sulphate

I gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

### Reducing Sugars

1 gram dissolved in 10 c.c. of water and boiled with 10 c.c. of Fehling's solution should not show any immediate reduction.

# SULPHANILIC ACID A.R.

 $C_6H_4$ .  $NH_2$ .  $SO_3H$  1:4 = 173·12

Maximum Limits of Impurities

Ash			0.02	per cent.
Chloride (Cl)			0.003	per cent.
Sulphate (SO <sub>2</sub> )			0.01	per cent.

White or cream-coloured crystals, slightly soluble in cold water, almost insoluble in alcohol.

#### Ash

2 grams should not leave more than 1 milligram of residue on ignition.

### Chloride

1 gram dissolved in 20 c.c. of hot water and acidified with 1 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

1 gram dissolved in 20 c.c. of hot water should not show any turbidity on addition of barium chloride solution.

### Assay

Titrate 5 grams suspended in water against N/1 NaOH, using phenolphthalein as indicator.

• 1 c.c. N/1 NaOH  $\equiv$  0 · 1731 gram  $C_6H_4$  · NH<sub>2</sub> · SO<sub>3</sub>H

Not less than 99 per cent. should be indicated.

# SULPHURIC ACID

 $\mathbf{H_2SO_4} = 98 \!\cdot\! 08$ 

### Maximum Limits of Impurities

Non-volatile matter			not appreciable
Hydrochloric Acid (C	.)		0.0005 per cent.
Nitric Acid (N <sub>2</sub> O <sub>5</sub> )			0.00002 per cent.
Heavy Metals and Iron	. •		0.0002 per cent.
Selenium			no reaction
Oxygen absorbed (O)			o·ooo1 per cent.
A CATTERN			o·oo14 per cent.
Arsenic $(As_2 O_3)$ .		٠	o·ooooı per cent.

A clear, colourless, oily liquid.

### Specific Gravity

About 1.84.

#### Residue

10 grams evaporated in a platinum dish and ignited should not leave any residue.

### Hydrochloric Acid

2 grams diluted with 20 c.c. of water should not show any opalescence on addition of silver nitrate solution.

### Nitric Acid

Dilute 6 c.c. with 2 c.c. of water and add 1 drop of hydrochloric acid and 2 milligrams of diphenylamine. No blue colour should be produced.

(Continued overleaf)

#### Heavy Metals and Iron

20 grams diluted with 30 c.c. of water and made alkaline with ammonia should not show more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Selenium

To 2 c.c. of the acid add very carefully, so as to form a layer, 2 c.c. of hydrochloric acid containing a trace of sodium sulphite. No red colour should be produced.

### Reducing Substances

15 c.c. diluted with 60 c.c. of water and cooled should remain coloured for 5 minutes on addition of 0.05 c.c. of N/10 KMnO<sub>4</sub>.

#### Ammonia

Dilute 1 c.c. of the acid with 40 c.c. of water, add 2 grams of sodium hydroxide (free from ammonia) and 2 c.c. of Nessler's reagent. The colour produced should not exceed that given by 0-025 milligram of ammonia.

#### Arsenic

Limit 0.1 part per million.

Test as described on page 189 using 10 c.c. of the acid and a few drops of stannous chloride solution. No hydrochloric acid is required.

### Assay

Titrate about 2 grams diluted with water against  $\,N/1\,$  NaOH, using methyl red as indicator.

1 c.e. N/1 NaOH  $\equiv$  0.04904 gram H<sub>2</sub>SO<sub>4</sub>

Not less than 98 per cent. should be indicated.

# TARTARIC ACID

COOH.CHOH.CHOH.COOH = 150.05

### Maximum Limits of Impurities

Ash			•	•	•	•	0.05	per	cent.
Sulph	ate				•		0.01	per	cent.
Heavy	y Meta	als an	d Iror	1.			0.0005	per	cent.

Colourless crystals, readily soluble in water and in alcohol.

#### Ash

10 grams ignited in a platinum dish should not leave more than 2 milligrams of residue.

### Sulphate

1 gram dissolved in 20 c.c. of water should not show any turbidity on addition of barium chloride solution.

### Heavy Metals and Iron

5 grams dissolved in 40 c.c. of water and excess of ammonia added should produce an almost colourless solution, which should not give more than a faint darkening on addition of 1 drop of sodium sulphide solution.

#### Assay

Dissolve about 3 grams (previously dried at 100°) in water and titrate against N/1 NaOH, using phenolphthalein as indicator.

1 c.c. N/1 NaOH = 
$$0.07502$$
 gram  $C_4H_6O_6$ 

Not less than 99.5 per cent. should be indicated.

# TRICHLORACETIC ACID

 $CCl_3COOH = 163 \cdot 38$ 

### Maximum Limits of Impurities

Ash			0.01	per cent.
Chloride (Ol)			0.001	per cent.
Sulphate (SO <sub>3</sub> )			0.01	per cent.
Nitrate (NoOr)			0.003	per cent.

Deliquescent crystals, with a pungent odour; very soluble in water forming a clear, colourless solution.

10 grams should not leave more than 1 milligram of residue on ignition.

1 gram dissolved in 20 c.c. of water should not show any opalescence on addition of silver nitrate solution and nitric acid.

1 gram dissolved in 20 c.c. of water should not show any turbidity or precipitate on addition of barium chloride solution and 0.5 c.c. of hydrochloric acid.

(Continued overleaf)

#### Nitrate

1 gram dissolved in 10 e.e. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 e.e. of sulphuric acid.

### Assay

Titrate 2 grams dissolved in water against N/1 NaOH using phenolphthalein as indicator.

1 c.c. 
$$N/1$$
 NaOH  $\equiv 0.1634$  gram  $CCl_3COOH$ 

Treat 0.2 gram with anhydrous sodium carbonate as described under benzoic acid, page 39, adding 50 c.c. of N/10 AgNO<sub>3</sub>.

1 c.e. 
$$N/10 \text{ AgNO}_3 \equiv 0.005446 \text{ gram CCl}_3 COOH$$

Not less than 99 per cent. should be indicated.

## URANIUM ACETATE

 $\begin{array}{c} UO_2(C_2H_3O_2)_2.2H_2O=424\cdot 22\\ or\ UO_2(C_2H_3O_2)_2.3H_2O=442\cdot 23 \end{array}$ 

Maximum Limits of Impurities

Sulphate $(5O_3)$		•	•	0.01	per	cent.
Calcium (Ca)				0.005	per	cent.
Heavy Metals				0.001	per	cent.
Alkalis (including	g Calc	ium)		0.4	per	cent.

A bright yellow crystalline powder, soluble in water forming a clear or slightly opalescent solution which becomes clear on acidifying with acetic acid.

### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

### Calcium and Heavy Metals

1 gram dissolved in 20 c.c. of water acidified with acetic acid should not give any precipitate on addition of excess of ammonium carbonate solution, or any darkening on the further addition of 1 drop of sodium sulphide solution.

### Alkalis

Dissolve 1 gram in water, acidify with acetic acid, heat to boiling, precipitate with ammonia, and filter. The filtrate evaporated to dryness and ignited should not leave more than 4 milligrams of residue.

# URANIUM NITRATE A.R.

 $UO_9(NO_3)_2.6H_2O = 502.25$ 

### Maximum Limits of Impurities

Sulphate (SO <sub>3</sub> )				0.01	per	cent.
Calcium (Ca)				0.005	per	cent.
Heavy Metals				$\mathtt{0.001}$	per	cent.
Alkalis (including	χC	alcium)		0.4	per	cent.

Brilliant yellow transparent crystals, readily soluble in water, alcohol and ether.

### Sulphate

1 gram dissolved in 20 c.c. of water and acidified with 0.5 c.c. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

#### Calcium and Heavy Metals

1 gram dissolved in 20 c.c. of water should not give any precipitate on addition of excess of ammonium carbonate solution, or any darkening on further addition of 1 drop of sodium sulphide solution.

#### Alkalis

Dissolve 1 gram in 20 c.c. of water, heat to boiling, precipitate with ammonia, and filter. The filtrate, evaporated to dryness and ignited, should not leave more than 4 milligrams of residue.

# VANILLIN COLL A.R.

 $C_6H_3$ , OH, OCH<sub>3</sub>, CHO  $4:3:1=152\cdot06$ 

### Maximum Limit of Impurities

Alcohol-insoluble matter . . nil
Ash . . . . . o o o per cent.

White or cream coloured, small, acicular crystals, with an aromatic odour; readily soluble in alcohol forming a clear solution.

### Melting Point

81° to 82°.

(Continued overleaf)

#### Ash

2 grams should not leave more than  $0\cdot 2$  milligrams of residue on ignition.

### Assay

Dissolve 3 grams in alcohol and titrate with N/2 alcoholic KOH using phenolphthalein as indicator.

1 c.c. N/2 KOH = 0.07603 gram  $C_6H_3$ . OH. OCH $_3$ . CHO

Not less than 99 per cent. should be indicated.

# ZINC A.R. (GRANULATED)

 $Zn = 65 \cdot 38$ 

Special for Gutzeit Test

### Maximum Limits of Impurities

Small granular masses of a silver grey metal; completely soluble in dilute hydrochloric acid.

#### Iron

Dissolve 20 grams in a slight excess of hydrochloric acid, boil with a few drops of nitric acid, add excess of ammonia, filter through a Buchner filter of 4 c.m. diameter and wash with water.

Not more than a faint brown precipitate should be left on the paper.

### 1 1 ---

Limit 0·1 part per million.

Test as described on page 180 using 20 grams of the zine, 50 c.c. of hydrochloric acid, 250 c.c. of water and a few drops of stannous chloride solution.

### Sensitivity

When tested in a Gutzeit apparatus after the addition of 0.01 milligram of arsenic (As<sub>2</sub>O<sub>3</sub>) a normal stain should be produced.

### Oxidisable Impurities

Dissolve 10 grams in 50 c.c. of water and 15 c.c. of sulphuric acid in a flask from which air is excluded; when solution is complete, add 0.1 c.c. N/10 KMnO<sub>4</sub>. A permanent pink colour should be produced.

# ZINC OXIDE A.R.

ZnO = 81.38

### Maximum Limits of Impurities

Insoluble in dilut	e aci	d.		nil
*Chloride (Cl)				0.002 per cent.
Sulphate (SO <sub>3</sub> )				o or per cent.
Carbonate .				no reaction
Metallic Zinc		٠		none
Heavy Metals				o·oi per cent.
Arsenic (As <sub>2</sub> O <sub>3</sub> )				0.0005 per cent.

A white amorphous powder, insoluble in water but readily soluble in dilute mineral acids.

#### Chloride

1 gram dissolved in 50 c.c. of water and 3 c.c. of nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

### Sulphate

 $1~\rm gram$  dissolved in 50 e.e. of water and 4 e.e. of hydrochloric acid should not show any turbidity on addition of barium chloride solution.

### Carbonate and Metal Zinc

5 grams should dissolve in 50 e.c. of dilute hydrochloric acid containing 1 drop of lead acetate solution without effervescence and should form a clear solution free from any black particles.

### Heavy Metals

0.1 gram dissolved in 20 c.c. of water and 5 c.c. of acetic acid, and made alkaline with ammonia, should not show more than a slight brown colour on addition of 5 c.c. of dilute hydrogen sulphide water (one-tenth saturated).

### Arsenic

Limit 5 parts per million.

Dissolve 2 grams in 12 c.c. of brominated hydrochloric acid and 40 c.c. of water, add a few drops of stannous chloride solution and test as described on page 189.

### ZINC SULPHATE A.R.

 $ZnSO_4.7H_2O = 287.55$ 

### Maximum Limits of Impurities

Chloride (Cl)				0.001	per cent.
Nitrate (N <sub>2</sub> O <sub>5</sub> )				0.002	per cent.
Heavy Metals				0.002	per cent.
Iron (Fe) .				0.002	per cent.
Arsenic (As <sub>0</sub> O <sub>2</sub> )	_	_	_	0.0001	ner cent.

Colourless crystals, readily soluble in water forming a clear colourless solution which should not be acid to methyl orange.

#### Chloride

5 grams dissolved in 100 c.c. of water and acidified with nitric acid should not show more than a faint opalescence on addition of silver nitrate solution.

#### Nitrate

1 gram dissolved in 10 c.c. of water and 0.5 c.c. of indigo solution added should remain blue on addition of 10 c.c. of sulphuric acid.

### Heavy Metals

I gram dissolved in 30 c.c. of water, acidified with 2 c.c. of glacial acetic acid and then rendered alkaline with ammonia, should not show any immediate darkening on addition of 5 c.c. of dilute hydrogen sulphide water (one-tenth saturated).

#### Iron

2.5 grams boiled with 10 c.c. of water and 2 c.c. of nitric acid, cooled, and diluted to 50 c.c. with water, should not give a deeper colour on addition of 5 c.c. of ammonium thiocyanate solution than 0.05 milligram of iron treated in the same manner.

### Arsenic

Limit 1 part per million.

Dissolve 10 grams in 50 c.c. of water, add 10 c.c. of stannated hydrochloric acid and test as described on page 189.

### THE LIMIT TEST FOR ARSENIC

## Description of the Apparatus

The apparatus consists essentially of a glass tube, 200 millimetres long with an internal diameter of 6.5 millimetres; the upper end is cut off square and ground smooth, while the lower end is drawn out to a diameter of about 1 millimetre, and a hole about 2 millimetres in diameter is blown in the side of the tube where it is constricted.

The tube is fitted by means of a rubber bung into a wide-mouthed bottle of about 120 c.c. capacity in such a manner that the hole in the side of the tube is clear of the underside of the bung and the end of the drawn out portion is clear above the surface of the liquid in the bottle.

A piece of white filter paper,  $100 \times 50$  millimetres, which has been soaked in a solution of lead acetate and dried, is rolled up and placed in the tube so that the upper end is about 25 millimetres below the top of the tube. The function of the lead paper is to absorb traces of hydrogen sulphide from the issuing gases.

A piece of white filter paper, previously soaked in an aqueous solution of mercuric chloride and dried, is placed over the upper end of the tube in such a manner that the whole of the evolved gas passes through a circle of the paper 6-5 millimetres in diameter.



#### METHOD OF PERFORMING THE TEST

A weighed quantity of the substance is dissolved in 50 c.c. of water and 10 c.c. (or the specified amount) of stannated hydrochloric acid is added. This is placed in the wide-mouthed bottle; 10 grams of granulated zine is added, and the rubber stopper supporting the tube containing the lead acetate paper and fitted with the mercuric chloride paper is placed in position. The apparatus is allowed to stand in a warm place, such as on a hot plate, for about 40 minutes, but not exposed to direct sunlight.

The stain produced on the mercuric chloride paper is compared with standard stains produced in the same manner from known quantities of arsenic. The most useful standards for comparison are those produced by 0.002 mg., 0.004 mg., 0.006 mg., 0.008 mg., and 0.01 mg. of As<sub>2</sub>O<sub>3</sub>. These stains can be obtained by using from 0.2 c.c. to 1.0 c.c. of solution of arsenic (0.001 per cent. As<sub>2</sub>O<sub>3</sub> in 10 per cent. HCl). These stains fade on keeping and freshly prepared standards only should be used.

If the substance to be tested is insoluble in water, but soluble in hydrochloric acid, it is dissolved in sufficient stannated hydrochloric acid to leave an excess of 10 c.c. for reacting with the zinc, and the test is then carried out in the usual manner. If the substance is strongly alkaline and a vigorous reaction takes place on addition of acid, brominated hydrochloric acid is used to prevent any loss of arsenic, and the bromine is finally removed by the addition of a few drops of stannous chloride solution.

In some cases special treatment is necessary. This is detailed in full in the text.

The most accurate results are obtained when the stain produced corresponds to about 0.8 c.c. or 1.0 c.c. of the standard solution of arsenic, and the quantities of material tested should be varied so as to produce stains of about this magnitude.

Fresh lead paper should be used for each test, and the tubes should be kept dry and scrupulously clean. They should be cleaned before each experiment.

The mercuric chloride papers should be carefully preserved in a well-stoppered bottle and protected from light.

### REAGENTS FOR THE LIMIT TEST FOR ARSENIC

### SOLUTION OF STANNOUS CHLORIDE As T.

Stannous Chloride					33 grams
Hydrochlorie Acid					10 c.c.
Distilled Water, to p	rodu	ce .	_		100 c.e.

### SOLUTION OF BROMINE As T.

Bromine						10 c.c.
Potassium	Bron	nide				30 grams
Distilled V	Vater,	to pr	oduce			100 c.c.

### STANNATED HYDROCHLORIC ACID As T.

Hydrochloric acid containing 1 per cent. by volume of stannous chloride solution.

### BROMINATED HYDROCHLORIC ACID As T.

Hydrochloric acid containing 1 per cent. by volume of bromine solution.

### ARSENIC SOLUTION.

This solution must be freshly prepared. 1 c.c. == 0.01 milligram  $As_2O_3$ .

The complete Gutzeit test apparatus, together with 250 grams hydrochloric acid, 50 grams stannous chloride solution, 50 grams bromine solution, 100 grams granulated zinc, 15 grams solution of arsenic 1 per cent. (for dilution to make the standard arsenic solution), and 50 each lead and mercuric chloride papers, is supplied. For further particulars, see the B.D.H. Catalogue of Fine Chemical Products.

### REAGENT SOLUTIONS FOR GENERAL USE

The strengths of the various solutions used in the foregoing tests are as follows: —

#### Acetic Acid

300 grams per litre (approximately 5 N).

#### Ammonia

102 grams per litre (approximately 6 N).

### Ammonium Acetate

77 grams per litre (approximately N).

### Ammonium Carbonate

Ammonium Carbonate . . . . 40 grams
Ammonia solution (strong) . . . . 15 c.c.
Distilled Water, to produce . . . 1 litre
(approximately N).

### Ammonium Chloride

106 grams per litre (approximately 2 N).

### Ammonium Molybdate

### Ammonium Oxalate

 $24~{\rm grams}$  per litre (approximately 0.33 N).

### Ammonium Thiocyanate

76 grams per litre (approximately N).

### Barium Chloride

122 grams per litre (approximately N).

### Barium Nitrate

65 grams per litre (approximately 0.5 N).

### Cadmium Iodide

46 grams per litre (approximately 0.25 N).

#### Calcium Chloride

110 grams per litre (approximately N).

### Calcium Sulphate

A saturated aqueous solution.

### Copper Sulphate

125 grams per litre (approximately N),

### Ferric Chloride

54 grams per litre (approximately N).

### Ferrous Thiocyanate

Dissblve 2 grams of pure iron wire in 300 c.c. of 3.3 per cent. (w/y) air-free sulphuric acid. Decant the solution and mix with 60 c.c. of 10 per cent. air-free potassium thiocyanate. The reagent, which should be colourless, may be stored in an atmosphere free from oxygen.

### Hydrochloric Acid

About 35 per cent. HCl.

### Hydrochloric Acid, dilute

110 grams HCl per litre (approximately 3 N).

### Indigo

Dissolve indigo carmine in 20 per cent, sulphuric acid and standardise so that 10 c.c. is just decolorised by 10 c.c. of M/1000 KNO<sub>2</sub> and 20 c.c. of sulphuric acid.

# Lead Acetate

95 grams per litre (approximately 0.5 N).

### Lead Ammonio-citrate

Lead Acetate					1 gram
Citric Acid	• .				5 grams
Water .					90 c.c.
Ammonia solu	ution (	strong)			5 c.c.

#### Nitric Acid, dilute

126 grams  $\mathrm{HNO_3}$  per litre (approximately 2 N).

# m-Phenylene-diamine Sulphate

2.5 grams per litre.

### Phosphate Reagent A

A 2.5 per cent. w/v solution of ammonium molybdate in 5N  $\rm H_2SO_4$ 

### Phosphate Reagent B

1-Amino-2-naphthol-4-sulphonic acid	0∙5 gram
15 per cent. w/v solution of sodium meta- bisulphite	195 c.c.
20 per cent. w/v solution of sodium sulphite	5 c.c.

Should be freshly prepared and the crystals should be rinsed

100 grams per litre (approximately 1.5 N).

42 grams per litre (approximately 0.1 M).

166 grams per litre (approximately N).

56 grams per litre (approximately N).

with distilled water before dissolving.

Potassium Cvanide

Potassium Ferricyanide
1 per cent, solution.

Potassium Ferrocyanide

Potassium Iodide

Potassium Hydroxide

Potassium Plumbite
Lead Acetate

Potassium Citrate

Potassium Hydroxide .

Zinc Ammonium Chloride (M/10)

with ammonia and dilute to 1 litre.

Distilled Water, to produce .

### Silver Nitrate 51 grams per litre (approximately 0.3 N). Sodium Hydroxide 200 grams per litre (approximately 5 N). Sodium Phosphate 108 grams per litre (approximately 0.3 M). Sodium Sulphide 120 grams per litre (approximately N). Sulphuric Acid, dilute 100 grams per litre (approximately 2 N). Uranium Acetate 35 grams Uranium Acetate . Acetic Acid, glacial . 50 c.c. 1 litre Distilled Water, to produce .

Dissolve 6.538 grams of pure zinc in a slight excess of hydrochloric acid, add 5 grams of ammonium chloride, render alkaline

2.5 grams 5 grams

75 grams

150 c.c.

# **B.D.H. INDICATORS**

For the Determination of Hydrogen Ion Concentration



Over sixty indicators, covering a range of pH from -0.2 to 13.0, are available either in solid form or in solution. The B.D.H. Universal Indicator is a mixed indicator which is of great value for rapid determination of the approximate pH of a fluid. Much greater accuracy may subsequently be secured by the use of individual indicators suitable to the particular circumstances. The B.D.H. Comparator is extremely useful for determining the pH of substances of low buffer content such The B.D.H. Capillator is as natural waters. of particular application when only small amounts of material are available, or when the test solution is highly coloured or contains matter in suspension. Both the Comparator and Capillator are issued in compact and convenient forms, as well suited to the needs of the field worker as to those of his colleague in the laboratory.

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